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FIELD DEMONSTRATION FOR BIOREMEDIATION TREATMENT FINAL REPORT

TECHNOLOGY DEMONSTRATION OF SOIL VAPOR EXTRACTION OFF-GAS AT McCLELLAN AIR FORCE BASE

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FINAL REPORT: NOVEMBER 1997 TO APRIL 1998

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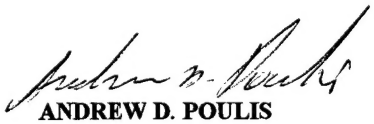

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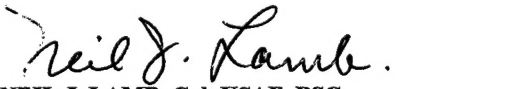

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13. ABSTRACT *(Maximum 200 words)*

McClellan Air Force Base (AFB) is a National Test Location designated through the Strategic Environmental Research and Development Program (SERDP), and was selected as the candidate test site for a demonstration of soil vapor extraction (SVE) off-gas treatment technology. A two-stage reactor system was employed for the treatment of the off-gas. The biological treatment was conducted at Operable Unit (OU) D Site S, located approximately 400 ft southwest of Building 1093. The SVE system at this area normally operates at a nominal volumetric flowrate of approximately 500 to 600 standard cubic feet per minute (scfm). The contaminated air stream from the SVE system that was fed to the reactor system operated at a flowrate of 5 to 10 scfm.

The demonstration of this reactor system started with the mobilization of field equipment and personnel in November 1997. The two-stage reactor system consisted of a fixed-film biofilter followed by a completely mixed (by continuous stirring), suspended-growth biological reactor. This reactor configuration was based on a review of the literature, on characterization of the off-gas from the SVE system being operated at McClellan AFB, and on the results of the laboratory study recently conducted by Battelle and Envirogen for this study (Battelle and Envirogen, 1997c).

The demonstration was run for approximately 18 weeks. This final demobilization and decommissioning of the pilot-scale reactor unit was completed on April 30, 1998.

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PREFACE

This report was prepared by Battelle, 505 King Avenue, Columbus, Ohio 43201, under Contract No. F08367-95-D-6004/DO 5401, for Air Force Research Laboratory, Airbase and Environmental Technology Division (AFRL/MLQ), 139 Barnes Drive, Suite 2, Tyndall Air Force Base, Florida 32403-5323. This reported work was funded by the United States Air Force.

This final report describes the Technology Demonstration of Soil Vapor Extraction Off-gas Treatment conducted at McClellan Air Force Base, California, the design of the off-gas treatment system, the modifications made to the system to improve the destruction removal efficiencies; the experimental methodologies used to monitor the technology performance; the data analysis techniques; a discussion of the significance of the experimental findings; and recommendations for future work in the application of this technology.

The work was performed between November 1997 and April 1998. The AFRL/MLQ project manager was Major Tim Wiley.

Contents

Contents
(Continued)

Contents
(Continued)

Contents
(Continued)

Figures

Tables

Abbreviations and Acronyms

AFB	Air Force Base
ARAR	applicable or relevant and appropriate requirements
AFRL/MLQE	Air Force Research Laboratory, Airbase and Environmental Technology Division
BCA	bicinchoninic acid
BFE/CSTRI	biofilter effluent/CSTR influent (gas stream)
BFI	biofilter influent (gas stream)
BOD	biological oxygen demand
BSM	Basal Salt medium
CAA	Clean Air Act
CAC	chlorinated aliphatic compound
cat ox	catalytic oxidation
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
cfm	cubic feet per minute
CFR	Code of Federal Regulations
Cl^-	chloride
CLP	Contract Laboratory Program
COC	chlorinated organic compound
COD	chemical oxygen demand
Cpost	effluent vapor concentration
Cpre	influent vapor concentration
CSTR	continuously stirred tank reactor
CSTRE	CSTR effluent
CWA	Clean Water Act
DCA	dichloroethane
DCB	dichlorobenzene
DCE	dichloroethene
DI	deionized
DL	detection limit
DoD	Department of Defense
DRE	destruction and removal efficiency
FID	flame ionization detector
FNPT	female national pipe thread
Fl	effluent liquid discharge rate
Fv	vapor flowrate
G-4	*Psuedomonas cepacia* G-4
G&A	general and administrative costs
GAC	granular activated carbon
GC	gas chromatography

gpd	gallon(s) per day
gph	gallon(s) per hour
HCl	hydrochloric acid
HP	Hewlett Packard
IDW	investigation-derived waste
IR	Installation Restoration
IWTP	industrial wastewater treatment plant
LWD	length, width, depth
MB	(system) mass balance
NA	not applicable
NC	not calculated
NCOC	nonchlorinated organic compound
ND	not detected
NEC	National Electric Code
NETTS	National Environmental Technology Test Site
NFPA	National Fire Protection Association
NIOSH	National Institute for Occupational Safety and Health
OD	optical density
OSHA	Occupational Safety and Health Act
OU	Operable Unit
PCE	tetrachloroethene
P&I	process and instrumentation
PID	photo ionization detector
PLFA	phospholipid fatty acid
PPE	personal protective equipment
ppmv	parts per million by volume
PQL	practical quantitation limit
psig	pounds per square inch gage
QA	quality assurance
QC	quality control
RCRA	Resource Conservation and Recovery Act
RE	removal efficiency
ROC	regulated organic compound
scfm	standard cubic feet per minute
SDWA	Safe Drinking Water Act
SERDP	Strategic Environmental Research and Development Program

SM-ALC/EMPC	Sacramento Air Logistics Center, Environmental Compliance Branch
SMAQMD	Sacramento Metropolitan Air Quality Management District
SOP	Standard Operating Procedure
SPCCP	Spill Prevention, Control, and Countermeasures Plan
SVE	soil vapor extraction
TCA	trichloroethane
TCLP	Toxicity Characteristic Leaching Procedure
TDS	total dissolved solids
TNMOC	total nonmethane organic carbon
TOC	total organic carbon
TOM	toluene *ortho*-monooxygenase
TSS	total suspended solids
TVS	total volatile solids
USC	United States Code
U.S. EPA	U.S. Environmental Protection Agency
VAC	voltage of alternating current
VC	vinyl chloride
VOA	volatile organic analysis
VOC	volatile organic compound
VSS	volatile suspended solids
w/w	weight by weight

1.0 Executive Summary

1.1 Background

McClellan Air Force Base (AFB) is a National Test Location designated through the Strategic Environmental Research and Development Program (SERDP), and was selected as the candidate test site for a demonstration of soil vapor extraction (SVE) off-gas treatment technology. The demonstration was managed by the Air Force Research Laboratory, Airbase and Environmental Technology Division (AFRL/MLQE) Tyndall AFB, Florida. A two-stage reactor system was employed for the treatment of the off-gas from the SVE system at McClellan AFB. The biological treatment was conducted at Operable Unit (OU) D Site S, located approximately 400 ft southwest of Building 1093. The SVE system at this area normally operates at a nominal volumetric flowrate of approximately 500 to 600 standard cubic feet per minute (scfm). The contaminated air stream from the SVE system that was fed to the reactor system operated at a flowrate of 5 to 10 scfm.

The demonstration of this reactor system started with the mobilization of field equipment and personnel in November 1997. The two-stage reactor system consisted of a fixed-film biofilter followed by a completely mixed (by continuous stirring), suspended-growth biological reactor. This reactor configuration was based on a review of the literature, on characterization of the off-gas from the SVE system being operated at McClellan AFB, and on the results of the laboratory study recently conducted by Battelle and Envirogen for this study (Battelle and Envirogen, 1997c).

The demonstration was run for approximately 18 weeks. The final demobilization and decommissioning of the pilot-scale reactor unit was completed on April 30, 1998.

1.2 Demonstration Description

The following objectives were defined for this demonstration:

- Determine the effectiveness of biological treatment processes for the treatment of off-gas from the full-scale SVE unit located at McClellan AFB.

- Quantify the total volatile organic compound (VOC) mass removed by the biotreatment process, and compare observed destruction and removal efficiencies (DREs) with the target DRE of 95%.

- Assess the performance of the treatment system during the demonstration.

- Obtain the data necessary to determine the cost to scale-up the technology, and the data necessary to support the design of the technology for full-scale application.

The first-stage bioreactor was a fixed-film biofilter, designed to remove VOCs that can be used as sources of carbon or energy by aerobic bacteria from the process-gas stream. This biofilter was a vertical reactor that contained a stable (i.e., previously composted) organic medium composed of wood chips and compost to support biomass growth. The biofilter operated with aqueous flow over the medium to facilitate contaminant mass transfer from the gas phase to the bacteria.

The biofilter medium was inoculated initially with a general inoculum (Phenobac®; Polybac Corporation, Bethlehem, Pennsylvania). The biofilter medium was not sterilized prior to being inoculated with the general inoculum, an approach consistent with general industry practice when working with biofilter media. Contaminated air entered the biofilter unit and flowed upward through the medium. All liquid discharged from the biofilter was pumped to a liquid waste storage container for later disposal by the Air Force.

Basic components of the system included the bioreactor, medium, control panel, and a vapor feed blower. The first-stage bioreactor was not fed a cosubstrate for cometabolic degradation of chlorinated aliphatic compounds (CACs), but was used to promote the degradation of growth-related contaminants in the off-gas. The removal of these contaminants resulted in a simplified gas stream that was fed to the second stage reactor.

The second-stage bioreactor, a continuously stirred tank reactor (CSTR), was used to degrade the remaining contaminants from the process-gas stream. The reactor was inoculated with *Pseuedomonas cepacia*, Strain G-4 (G-4), a bacterium provided by Envirogen, the contractor who performed installation and performance monitoring. Thus, the second stage was a cometabolic reactor that used phenol as the cosubstrate.

The field pilot CSTR was composed of the bioreactor vessel with automatic pH, foam, and temperature control (a heating element and a refrigerated cooling coil); nutrient feeds; and

discharge tanks. A blower delivered contaminated vapor to the bottom of the bioreactor. The vapor was then mixed vigorously with the liquid contents of the vessel to facilitate the growth of the degradative bacteria. As the vapor bubbled through the liquid column, trichloroethene (TCE), dichloroethene (DCE), and vinyl chloride (VC) were transported by molecular diffusion from the vapor to the liquid where they were destroyed with limited success by the bacteria. The treated vapor exited the liquid at the top of the water column, and was discharged from the reactor into two in-series carbon canisters to completely remove any remaining organic contaminants. A wastewater holding tank was used to allow for batch disposal of wastewater following analysis at an off-site laboratory.

1.3 Results

As stated, the CSTR was inoculated with G-4 bacteria, which has been reported in the literature to actively degrade chloroethene compounds (Battelle and Envirogen, 1997a). G-4 also has been used successfully by Envirogen (DOE, 1994) for TCE degradation. However, in this demonstration, the 95% target DRE was not achieved, even after the G-4 bacteria were reinoculated to the CSTR on two separate occasions.

Total DREs for each reactor component ranged from 43 to 73% in the biofilter and from zero to 15% in the CSTR. Total system DREs ranged from 51 to 74%, which did not meet the target DRE of 95%.

1.4 Conclusions

Although biofilter removal efficiencies were high, the entire two-stage reactor system did not meet the target 95% DREs. The first-stage biofilter performed as expected by producing high DREs for nonchlorinated compounds, as was the case for toluene. DREs for chlorinated compounds were higher than expected in the biofilter. The removal mechanism for the chlorinated compounds in the biofilter remains unclear but could be attributed in part to sorption or to biological transformation.

In spite of its success at other AFB sites (e.g., DREs of 74% were observed at the Robins AFB, Georgia [Guarini and Folsom, 1996] and DREs of 85-90% were observed at F.E. Warren

AFB, Wyoming [Radian International, 1996]), the CSTR DREs were much lower than expected and did not meet the performance criteria of 95% contaminant destruction. Laboratory shake-flask tests were conducted by Envirogen to determine whether site water or site chemicals and nutrients could have an inhibitory effect on the G-4 bacteria. Results indicated that distilled water outperformed site water with respect to G-4 growth and TCE degradation. However, a water purification system installed at the site provided no noticeable improvement in G-4 growth or TCE degradation at the site.

Laboratory investigations showed that the inoculated G-4 population was outgrown by an unidentified, non-TCE-degrading, phenol-degrading culture. These results suggest that G-4 was unstable under the McClellan AFB site conditions.

1.5 Recommendations

The two-stage reactor system demonstrated at McClellan AFB, which includes the biofilter plus CSTR, is not recommended for treatment of SVE off gases at McClellan AFB. The combined system was not successful in destroying 95% of the VOCs in the SVE off-gas stream. For reasons that could not be determined during this study, the CSTR DREs were much lower than expected, and the total system performance criterion of 95% was not met.

2.0 Introduction and Background

This section introduces the United States Department of Defense (DoD) National Environmental Technology Test Site (NETTS) program at McClellan AFB, where the treatment technology demonstration took place. The treatment technology involved the ex situ biological treatment of the off-gas from the existing SVE treatment system. This introduction includes an overview of the NETTS program, a discussion of the general need for innovative environmental cleanup, characterization, and monitoring technologies, a description of McClellan AFB, a brief description of the bioremediation of SVE off-gas, and a presentation of the scope of work performed during the technology demonstration.

2.1 Strategic Environmental Research and Development Program (SERDP) and the National Environmental Technology Test Site (NETTS) Program

McClellan AFB is a National Test Location designated through SERDP. The NETTS mission is to provide locations for the comparative demonstration and evaluation of innovative technologies performing environmental characterization, cleanup, and monitoring. Candidate innovative technologies must have the potential to be more cost-effective or to achieve significantly reduced risks than currently available processes. The technologies also must be applicable to Installation Restoration (IR) efforts at DoD facilities. The ultimate goal of NETTS is to accelerate the market availability of these new DoD-applicable technologies.

2.2 Technology Objectives

This section describes the objectives of the technology demonstration performed at McClellan AFB. Four objectives were defined for this project. These objectives are detailed as follows:

- Determine the effectiveness of the biotreatment processes for the treatment of SVE off-gases containing halogenated and nonhalogenated organic compounds.

- Quantify the total VOC mass removed by the process and compare observed DREs with the target DRE of 95%.
- Assess the treatment system performance.
- Obtain the data necessary to determine the cost to scale up the technology.

Data needs, data acquisition requirements, and data interpretation issues are described in detail for each objective in the following subsections. These data criteria were developed on the basis of current site information provided to Battelle.

2.2.1 Objective 1: Remediation Effectiveness

The effectiveness of the biological treatment technology for remediation of the McClellan AFB off-gas stream was measured as: (1) the degree to which the contaminants in the off-gas stream were destroyed; (2) the degree to which the vapor flowrate through the biological reactors was maximized; (3) the degree to which the cosubstrate addition rate to stage 2 of the process was minimized; and (4) the degree of contaminant removal over time. Success in reaching specified DREs was measured by the levels of residual VOC contaminants remaining in the process effluent off-gas and wastewater.

2.2.1.1 Data Needs

For this study, McClellan AFB defined an overall cleanup requirement of 95% VOC destruction, based on Sacramento Metropolitan Air Quality Management District (SMAQMD) regulations. The 95% DRE requirement was based on the destruction of the VOCs in the off-gas process stream, and not on the destruction of each individual contaminant. Furthermore, the overall DRE reflects the mineralization of the contaminant's organic carbon, not merely the destruction or modification of the original contaminant structure (e.g., biological transformation of a substituted phenol compound to a substituted catechol compound).

Remediation effectiveness was measured by the degree to which 95% DREs were met. The technology remedial effectiveness was evaluated on the basis of contaminant concentration reductions in the gas phase within the stages of the biotreatment system and as a function of vapor flowrate and cosubstrate addition rate. Data required for the remedial effectiveness

assessment included influent and effluent vapor contaminant concentrations from the reactor stages, vapor flowrates, and cosubstrate addition rates. Because the CSTR liquid medium was discharged continually, liquid samples were collected and analyzed for residual contaminant concentrations, biological oxygen demand (BOD), and chemical oxygen demand (COD) to ensure that an overall mass balance was being obtained, and to quantify the amount of VOCs, BOD, and COD discharged with the wastewater stream. The wastewater also was monitored for pH and chloride production to control pH and to regulate the amount of pH-buffer that was added to the system. Total suspended solids (TSS) and total dissolved solids (TDS) were measured to monitor the biological growth in the CSTR. Waste streams were collected and characterized to determine the physical waste characteristics for disposal.

2.2.1.2 Data Acquisition

Influent and effluent vapor grab samples for contaminant concentration characterization were collected periodically and analyzed for individual contaminants. Grab samples of the liquid discharge from the reactor stages also were collected periodically and analyzed for individual contaminants. Sampling locations are described in Section 4.3.4. Vapor flowrates and cosubstrate addition rates were routinely monitored. In addition to vapor and liquid grab samples, an on-site gas chromatograph (GC) was used to sample the influent and effluent vapor streams from the reactor stages in order to evaluate the DREs of the targeted contaminants.

A hand-held photoionization detector (PID) was used to assess overall DREs by routinely analyzing the overall system influent and effluent total vapor VOC concentrations. The PID was not capable of not detecting methane; however, methane was not expected to be degraded significantly by either the biofilter or the CSTR because of its high Henry's law constant and vapor pressure. Methane also was not expected to interfere with the degradation of other compounds in the process gas stream. The PID preferentially responded to compounds with double carbon-carbon bonds, including chloroethenes and toluene, the primary target compounds of this study. Influent and effluent grab samples also were collected periodically for U.S. EPA TO-12 analyses of total nonmethane organic carbon compounds and for U.S. EPA TO-14 analyses of VOC constituents to determine system DREs.

2.2.1.3 Data Analysis and Interpretation

Effluent vapor sampling results were performed to quantify the residual concentration of contaminants remaining in the treated process off-gas stream. These concentrations were compared with influent concentrations to determine whether the cleanup goals were being achieved. Effluent off-gas and residual contaminant concentrations in the liquid discharge were compared with influent process-gas VOC concentrations to establish a mass balance of contaminants in the system. Total and soluble VOC analyses were conducted on the liquid wastestream. DREs were monitored using on-site and off-site analyses of the influent and effluent process gas streams. For the duration of the field study, on-site analyses were conducted to monitor the process in the field and off-site analyses were used to assess process performance.

2.2.2 Objective 2: Contaminant Mass Quantification

The objective of contaminant mass quantification was to document the total amount of VOCs degraded during the demonstration. This section presents the methods used for this assessment.

2.2.2.1 Data Needs

The following data were needed to quantify the total mass of VOCs removed:

- Volume of air processed through the system during operation
- Measurements of influent and effluent vapor VOC concentrations
- Measurements of the vapor throughput rates
- Total mass of VOCs through the system during operation
- Measurements of effluent liquid contaminant concentrations
- Measurements of daily liquid waste discharge rate
- Total mass of VOCs discharged in the liquid wastestream.

The following target-compounds were monitored in the field and also were used to assess the performance of the biotreatment process:

- TCE
- Tetrachloroethene (PCE)
- 1,1,1-Trichloroethane (1,1,1-TCA)

- 1,2-*cis*-DCE
- 1,1-Dichloroethane (1,1-DCA)
- VC
- Toluene.

2.2.2.2 Data Acquisition

During the demonstration, influent and effluent vapor flowrates and liquid discharge rates from the reactor were monitored routinely. In addition, effluent liquid samples and influent and effluent samples of process gases were collected periodically to measure VOC concentrations in accordance with the sampling plan outlined in the project work plan (Battelle and Envirogen, 1997b).

After completion of the process, the system was disconnected and decommissioned. Accumulations of sediments, free liquid, biomass, and other residuals containing contaminant compounds were recovered. These residuals were analyzed to determine residual contaminant concentrations and to quantify residual waste disposal requirements.

2.2.2.3 Data Analysis and Interpretation

Mass quantification was performed by calculating the total mass removed from the influent vapor stream, and the total mass remaining in effluent vapor stream and the effluent liquid stream. The total mass of contaminants removed from the influent vapor stream was determined using estimated removal rates calculated during specific sampling intervals done during the demonstration.

The removal rate of contaminants during the test was calculated periodically by measuring influent and effluent vapor flowrates and concentrations, and average liquid discharge rates and liquid concentrations. The cumulative mass removed was estimated by plotting these data versus time.

The total mass of contaminants in process residuals was determined using the mass and concentration data generated during decommissioning.

Removal efficiencies (REs) for the process gas stream also were calculated using the following equation:

$$RE = \left(1 - \frac{C_{post}}{C_{pre}}\right) * 100\% \qquad \text{(Equation 2-1)}$$

where: RE = process gas removal efficiency.

C_{post} = effluent vapor concentration (mg/m^3)

C_{pre} = influent vapor concentration (mg/m^3)

The system mass balance (MB) was calculated for each compound and for the overall VOCs in the reactor process by the following equation:

$$MB = \left[C_{post}/C_{pre} - (0.093)(F_l C_l)/(F_v C_{pre})\right][100\%] \qquad \text{(Equation 2-2)}$$

where: MB = system mass balance

C_l = effluent liquid concentration (mg/L)

F_l = effluent liquid discharge rate (gallons per day [gpd])

F_v = vapor flowrate (cubic feet per minute [cfm])

0.093 = conversion factor for: $\frac{gpd * mg/L}{cfm * mg/m^3}$

Based on these indices for the target compounds, an interpretation was made for the total percentage of contaminant degraded. This interpretation took into account the chemical and physical properties of the individual compounds and the operational parameters of the field test (e.g., duration and flowrates).

2.2.3 Objective 3: Treatment System Performance

The objective of monitoring treatment system performance was to document the performance of the biotreatment filter system during the demonstration. Performance was assessed in terms of effectiveness, efficiency, and integrity. This section explains how the assessment was performed.

2.2.3.1 Data Needs

To determine the treatment system performance, specific data were required from each of the two bioreactor stages used during the evaluation. The following data were collected for each bioreactor stage:

- Influent and effluent vapor flowrates, temperatures, pressures, and contaminant concentrations
- Liquid effluent discharge rate and contaminant concentrations
- Documented problems, failures, and downtime
- Utility usage, including measurements of makeup water and electricity
- pH fluctuations and amount of buffer used to control pH
- Nutrient amendment rates.

2.2.3.2 Data Acquisition

Two separate skid-mounted units were connected in series for the two-stage system. During the demonstration, performance data were collected manually from each bioreactor stage. The manual data readings were recorded on log sheets that were maintained in logbooks at the site. Copies of the logbooks are included in Appendix C. As much as possible, the following measurements were coordinated with process gas sampling events: process gas influent and effluent flowrates, temperature, pressure, liquid flowrates, and liquid temperatures. Data listed in Table 2-1 for biofilter unit and CSTR parameters were logged manually on a daily basis, except during periods of operational downtime and CSTR reinoculation (i.e., batch mode operation).

Alarms. In addition to manual data collection, the CSTR was equipped with the following alarms:

- High blower differential pressure alarm (On/Off)
- High blower temperature alarm (On/Off)
- Low pH alarm (On/Off)
- High pH alarm (On/Off)
- Low level alarm (On/Off).

Components: The CSTR was equipped with the following components:

- Blower (On/Off)
- Mixer (On/Off)
- Liquid heater (On/Off)
- Refrigeration pump (On/Off).

Table 2-1. Log Sheet Parameters Recorded for the Two-Stage Reactor System

Biofilter	CSTR
Feed gas inlet humidity (%)	Tank volume (gal)
Feed gas inlet temperature (°C)	Nutrient pump (%/stroke)
Feed gas inlet pressure (in. H_2O)	Nutrient pump (strokes/minute)
Feed gas flowrate (scfm)	Make-up water timer setting
Biofilter packing pressure drop (in. H_2O)	Make-up water totalizer reading (gal)
Upper hose timer setting	Inlet air flowrate to CSTR (scfm)
Lower hose timer setting	Outlet air flowrate from CSTR (scfm)
Upper hose totalizer reading (gal)	Inlet air temperature to CSTR (°C)
Lower hose totalizer reading (gal)	Inlet air pressure (psi)
Biofilter drain water pH	Caustic tank volume (gal)
Inlet vapor total hydrocarbon concentration (parts per million by volume [ppmv] benzene equivalents)	Caustic feed pump (%/stroke)
	Caustic feed pump (strokes per minute)
Intermediate vapor total hydrocarbon concentration (ppmv benzene equivalents)	Phenol tank volume (gal)
	Phenol feed pump (%/stroke)
Outlet vapor total hydrocarbon concentration (ppmv benzene equivalents)	Phenol feed pump (strokes per minute)
	pH Meter reading
	CSTR reactor liquid level
	Wastewater storage tank volume (gal)
	Bioreactor temperature (°C)

Notations of process problems, durations, and resolutions were recorded, including downtime, maintenance, modifications, and routine maintenance events.

2.2.3.3 Data Analysis and Interpretation

Data were analyzed and interpreted to evaluate system performance. The data were used to improve upon the operation and design of future treatment systems. Specific parameters that were calculated include the following:

- VOC removal from the process gas stream
- VOC concentrations in the liquid waste streams
- Makeup water requirement of the biotreatment system
- Electricity usage of the biotreatment system (although the electricity usage observed during the demonstration was not used for scale up)
- Pressure drop across the packing material within the biotreatment columns

- Percentage of operation period the system was off line as a result of shutdowns or required maintenance.

2.2.4 Objective 4: Cost Information

An important goal of the technology demonstration, secondary only to the evaluation of overall process effectiveness, was the determination of full-scale system costs. The ultimate cost of implementing and operating a full-scale system, based on the results of this demonstration, had a significant effect on the acceptability of the two-stage reactor system as a viable treatment option. An objective of the demonstration, therefore, was to generate, compile, and evaluate data needed to estimate the cost of implementing and operating a full-scale off-gas biological treatment system at McClellan AFB.

2.2.4.1 Data Needs

The data from the field-pilot evaluation that was needed to assess the scale-up cost of a full-scale system included the following:

- Raw materials usage, including inorganic nutrients, caustic, and cosubstrate
- Installation costs
- Water usage
- Electricity usage, although direct scaling of electrical costs was not appropriate
- Vapor pressure head losses in each of the reactors
- Vapor contact time requirements
- Routine laboratory analyses required for maintenance and operation
- Operation and maintenance
- Materials.

2.2.4.2 Data Acquisition

Cost data were generated and recorded as needed during all demonstration-related activities. The data were managed for the primary goal of calculating the full-scale system cost estimate, which includes breakdowns of cost items such as materials and utilities. All expenses were recorded and coded for tracking purposes.

2.2.4.3 Data Analysis and Interpretation

This technology demonstrated VOC DREs in the CSTR process insufficient to meet the 95% target DRE. The CSTR was designed to destroy the chlorinated organic contaminants in the SVE off-gas. For this reason, full-scale costs were not estimated. However, detailed costs for the pilot scale treatment demonstration are included in Section 7.

2.3 Technology Overview

The scope of work performed during this demonstration included the mobilization of the pilot unit to the site, the installation of pilot reactor(s), implementation of the pilot demonstration, demobilization from the site, and disposal of generated wastes. The major tasks completed for this scope of work are described below.

Prior to the field demonstration, a review of the SVE off-gas stream characteristics was conducted along with a literature review of possible treatment scenarios (Battelle and Envirogen, 1997a). Based on this review, it was determined that the most appropriate biocatalyst/bioreactor combination for the McClellan application was a two-stage biotrickling filter configuration using G-4 in the second stage reactor. A laboratory-scale demonstration was conducted for corroboration. After four months of laboratory testing, acetone and toluene DREs were greater than 90% and 80%, respectively. However, poor DREs (less than 5%) were observed for chlorinated VOCs using a synthetic air stream containing the major SVE target contaminants TCE, PCE, 1,1,1-TCA, and 1,2-dichlorobenzene. Because of the poor laboratory results attained using the two-stage biotrickling filter configuration, McClellan AFB, Battelle, and Envirogen agreed to use an alternate configuration for the field demonstration (i.e., a biofilter followed by a CSTR in series). Both of these technologies have been successfully applied independently, the biofilter for readily biodegradable hydrocarbons, and the CSTR for TCE treatment (Guarini and

Folsom, 1996; Radian International, 1996). The McClellan AFB demonstration was the first attempt at applying these two technologies in combination.

A laboratory-scale demonstration of the off-gas biofilter technology was conducted prior to the McClellan AFB field demonstration. Results of the demonstration were used to scale the field demonstration unit. Those results and the scale-up modifications were submitted in a report following the completion of the laboratory demonstration (Battelle and Envirogen, 1997c).

Additional laboratory studies were performed at Envirogen's laboratories during the field demonstration, to examine operational parameters that directly affected system performance. The additional laboratory experiments were performed at Envirogen laboratories, and the results of these additional laboratory tests are described in detail in Section 5 of this report.

The pilot reactors were mobilized to the field in November 1997, and installed by Battelle and Envirogen technicians and project personnel. The units were connected directly to the SVE off-gas manifold, immediately upstream of the existing off-gas treatment unit. Effluent from the biotreatment system was discharged to the atmosphere through activated carbon.

2.4 Demonstration Scope

The biotreatment system was tested to determine the overall DRE. The DREs of individual contaminants in the off-gas, the cosubstrate requirements, the stability of the reactor over the 18-week testing period, and the maximum vapor throughput were measured. Sampling was performed at the influent and effluent ports and between the two reactor stages to quantify the concentration of contaminants entering and exiting each reactor stage.

2.5 Document Organization

This report is organized according to the October 1996 NETTS Table of Contents outline for Technology Demonstration Application Analysis reports. Section 1, Executive Summary, provides a brief overview of the technology demonstration background, description, results, conclusions, and recommendations. Section 2, Introduction and Background, describes the NETTS Program and its objectives, and provides an overview, scope, and document organization. Section 3 provides site descriptions, and Section 4 describes the technology demonstration.

Section 5 presents results of the technology/performance evaluation. Section 6 presents other technology issues, including regulatory requirements, health and safety considerations, and community acceptance. A cost summary of the pilot demonstration is presented in Section 7, including the cost analysis basis, cost categories, and the actual costs. Recommendations and conclusions are given in Sections 8 and 9, respectively. Bibliographic data for references cited in the text and appendices are given in Section 10.

Six appendices support the material in the text. Appendix A provides the Phosolipid Fatty Acid Analysis report for the biofilter medium. Appendix B includes the Quality Assurance/Quality Control (QA/QC) report for the study. Field log sheets are provided in Appendix C, and on-site GC data is provided in Appendix D. Off-site analytical data for vapors and liquids are provided in Appendices E and F, respectively. Biomass sample analytical data is included in Appendix G, and biofilter PID and assay test data sheets are included in Appendix H.

3.0 Site Description

3.1 Location and Setting

McClellan AFB is located approximately 7 miles north of Sacramento, California. A general vicinity map is shown in Figure 3-1 (McClellan AFB, 1998). In July 1987, McClellan AFB was placed on the National Priorities List. McClellan AFB is divided into 11 OUs, defined as OUs A through H, B1, C1, and GW. The biological treatment demonstration was conducted at OU D, Site S, located approximately 400 ft southwest of McClellan AFB Building 1093 (Figure 3-2).

3.2 Geology

Not applicable for this project.

3.3 Hydrogeology

Not applicable for this project.

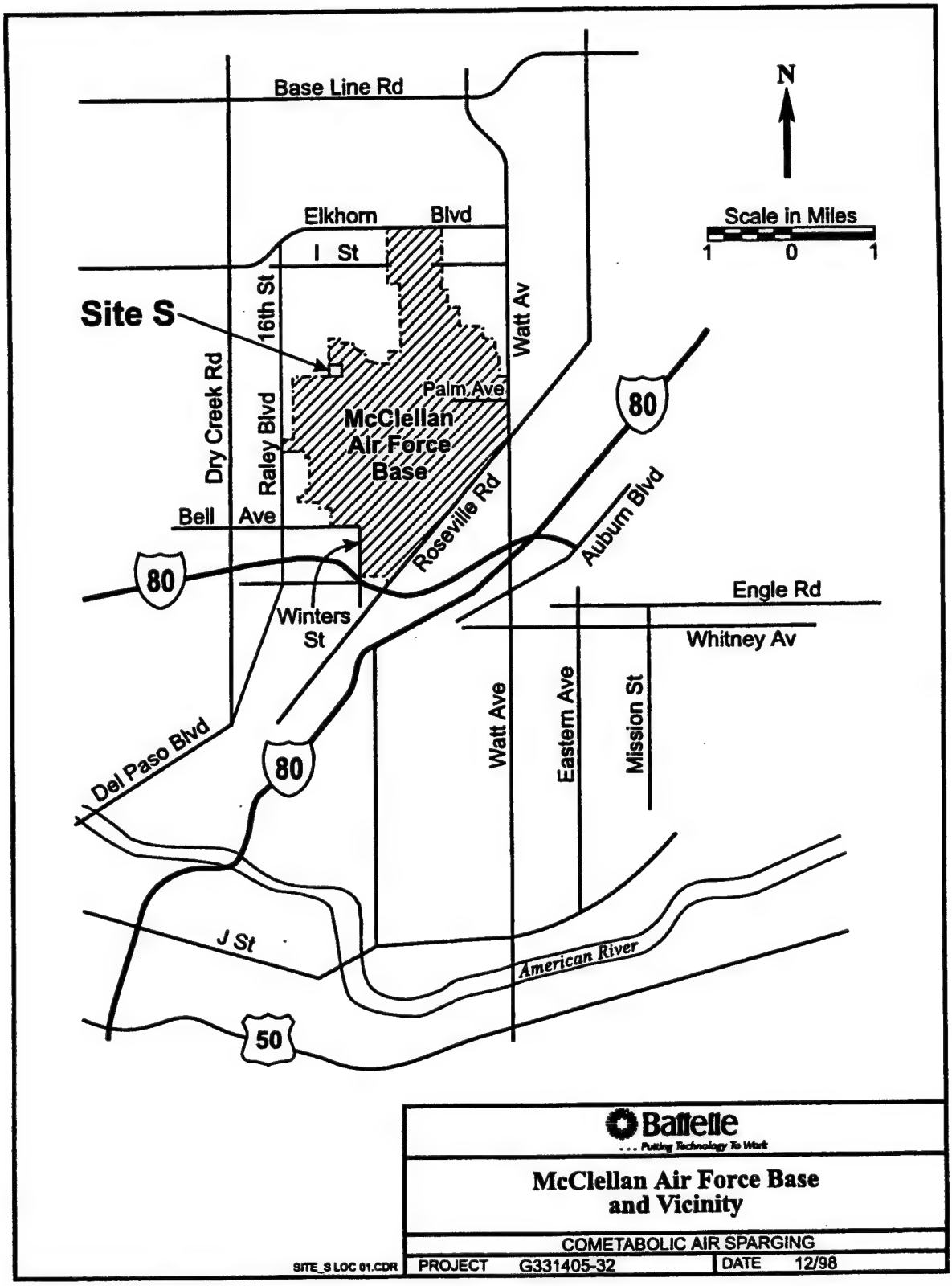

Figure 3-1. McClellan Air Force Base and Vicinity

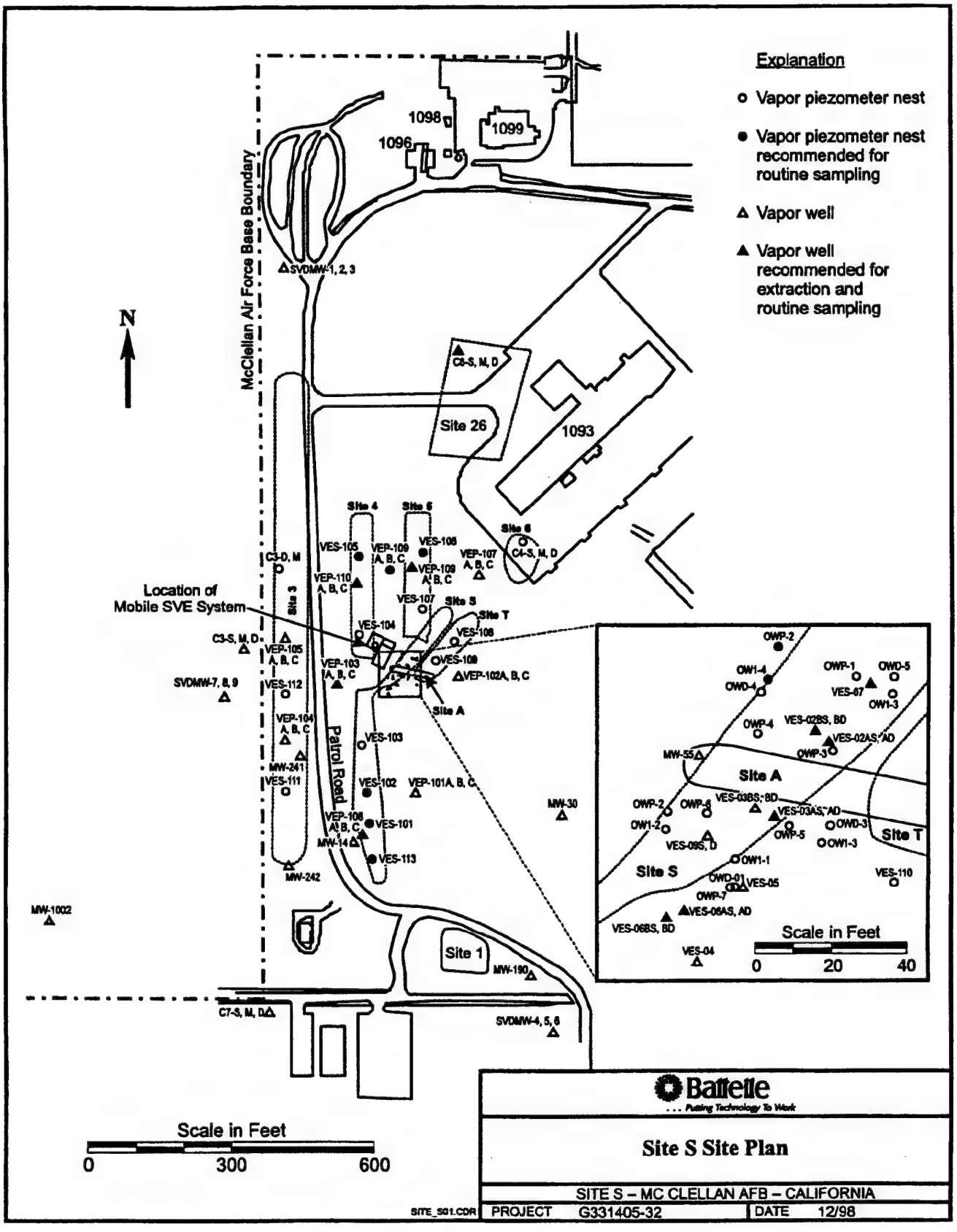

Figure 3-2. Site S Site Plan

3.4 Contaminant Distribution

The full-scale SVE system installed at OU D included 27 SVE wells, 39 piezometers, 5 vacuum pumps, and a catalytic oxidation (cat-ox) system followed by an acid scrubber system. The SVE wells are connected to a manifold system through which the extracted vapors are conducted to an air-water separator. The system was operated normally at a nominal volumetric flowrate of approximately 500 to 600 scfm during the demonstration.

The pilot treatment unit operated at flowrates of 5 to 10 scfm. Based on information provided by the Air Force, the expected total VOC concentration at OU D was 374 ppmv of chlorinated organic compounds (COCs) and 247.6 ppmv nonchlorinated organic compounds (NCOCs). The primary compounds of interest are listed in Table 3-1. These concentrations were measured during the demonstration by the on-site field personnel.

Table 3-1. Principal Compounds of Interest

Compound	Influent Concentration Range (average) (ppmv)
Trichloroethene	6.40 – 18.00 (14.90 ± 4.38)
Tetrachloroethene	3.50 – 22.00 (17.75 ± 7.06)
1,1,1-Trichloroethane	6.60 – 34.00 (26.30 ± 9.97)
1,2-*cis*-Dichloroethene	1.00 – 3.80 (2.07 ± 1.12)
1,1-Dichloroethane	1.60 – 3.80 (3.02 ± 0.82)
Vinyl chloride	0.23 – 1.20 (0.63 ± 0.42)
Toluene	4.10 – 9.30 (7.57 ± 1.83)

4.0 Demonstration Description

This section provides a detailed description of the bioreactor filter technology introduced in Section 1. The description includes a presentation of the principles, applicability, advantages, disadvantages, and status of the technology.

4.1 Technology Principles

A two-stage reactor system was used to treat of the SVE off-gas at McClellan AFB. The two-stage reactor system consisted of a fixed-film biofilter followed by a completely mixed, suspended growth biological reactor.

The first-stage biofilter was a fixed-film bioreactor, designed to remove VOCs that were used by the bacteria as sources of carbon or energy from the process-gas stream. The biofilter relied on the beneficial action of naturally occurring microorganisms to metabolize gas stream pollutants to harmless by-products such as carbon dioxide, water, and mineral salts. The first-stage biofilter was a vertical reactor that contained a stable (i.e., previously composted) organic medium comprised of wood chips and compost to support biomass growth. Water was added to the medium periodically to facilitate contaminant mass transfer from the gas phase to the aqueous phase and to support the growth of a biological film (biofilm) on the medium. The biofilm was responsible primarily for the degradation of the contaminants from the gas streams that do not require the addition of a cosubstrate for cometabolic degradation. The gas stream was introduced into the biofilter reactor at a counter-current flow to the liquid stream.

The biofilter media was inoculated initially with a general inoculum (Phenobac®; Polybac Corporation, Bethlehem, Pennsylvania) on December 6, 1997 after the completion of abiotic tests. Contaminated air entered the biofilter unit and flowed upward through the medium. The medium compartment was constructed with two sets of irrigation hoses to maintain 60 to 70% medium moisture levels through the addition of water at timed intervals. One set of hoses was positioned at the top of the bed, approximately 6 inches below the medium surface; and a second set of hoses was positioned at the bottom of the bed, between the medium and a lower humidification zone, which contained an inert layer of sorbent material (U.S. Patent 5,445,660). In addition to providing moisture, the upper irrigation hoses were used to periodically provide

inorganic soluble nutrients (nitrogen and phosphorus) to the medium via a slowly dissolving tablet. The lower humidification zone ensured that the air entering the biologically active area of the biofilter was saturated with water vapor. All liquid discharged from the biofilter was pumped to a liquid waste storage container for disposal.

Basic system components of the biofilter included the bioreactor, medium, control panel, and a vapor feed blower. The first stage biofilter was not fed a cosubstrate for cometabolic degradation of CACs, but was used to promote the degradation of growth-related contaminants in the off-gas. The removal of these contaminants was expected to result in a simplified gas stream that would be fed to the second stage reactor.

The second-stage CSTR was used to degrade the residual contaminants in the gas stream following treatment in the first-stage reactor. Principal contaminants included the chlorinated solvent compounds found in the contaminated stream that required a cosubstrate for cometabolic degradation. The second-stage CTSR was a cometabolic reactor, where phenol was used as the cosubstrate. The reactor was inoculated with G-4, grown at Envirogen's laboratory.

The field pilot CSTR was composed of the bioreactor vessel with automatic pH, foam, and temperature control (a heating element and a refrigerated cooling coil), nutrient feeds, and discharge tanks. A blower delivered contaminated vapor to the bottom of the CSTR. The vapor was then mixed vigorously with the vessel's liquid contents to facilitate biological growth, contaminant mass transfer, and VOC degradation. As the vapor bubbled through the liquid column, TCE and other chlorinated contaminants (e.g., DCE and VC) were transported via molecular diffusion from the vapor to the liquid where they could be taken up by the G-4 bacteria and cometabolized by the toluene *ortho*-monooxygenase (TOM) enzyme.

The treated vapor exited the liquid at the top of the water column and was discharged from the reactor. Nutrients and phenol, the growth substrates G-4, were metered into the reactor with a small volume of make-up water (approximately 5 gal per day). Wastewater was discharged from the reactor after passing over an overflow weir. The wastewater contained some residual biomass and salts that were discharged to a 1,000-gal storage tank. The 1,000-gal tank was used for batch disposal of wastewater into the headworks of the industrial waste treatment plant (IWTP), following off-site analysis to determine contaminant concentrations.

The CSTR was inoculated with G-4, which had been demonstrated in previous studies to actively degrade chloroethene compounds (for a review of the literature, see Battelle and

Envirogen, 1997a). G-4 expresses the TOM enzyme, which cometabolically degrades TCE and some other chloroethenes. The TOM enzyme is inducible by toluene, phenol, and *o*- or *m*-cresol (Nelson et al., 1986; Nelson et al., 1988). The biodegradation of TCE by G-4 in the prsence of phenol was investigated by Folsom et al. (1990), who found that the maximum degradation rate of G-4 was 1.9 mg TCE per mg of cell protein per day (mg/mg/day). Folsom and Chapman (1991) found that the maximum TCE degradation potential of G-4 was 1.1 mg/mg/day. Both studies showed that the concentration of phenol must be kept low to observe maximum TCE degradation rates and limit competitive inhibition effects.

G-4 also has been used successfully used by Envirogen (U.S. DOE, 1994) for TCE degradation. Through internal and external funding from the U.S. Departments of Energy and Defense, Envirogen has developed biological reactor-based treatment systems for the cometabolic destruction of TCE. Initially, research and development activities centered on gaining a thorough understanding of the biochemistry of TCE degradation, and selecting the most appropriate organisms to catalyze the degradation of chlorinated solvents. The development program continued through the construction of a novel suspended growth laboratory-scale continuous-stirred tank (bio)reactor, or CSTR. After extensive testing of the laboratory-scale unit (Ensley and Kurisko, 1994), Envirogen engineered and constructed a 4,000 L pilot-scale CSTR. Greater than 90% TCE removal has been demonstrated using the suspended growth design when treating a TCE/benzene stream (Folsom, 1992), when treating a wastestream containing other chlorinated solvents (DOE, 1994), and when treating a pure TCE wastestream at concentrations up to 5 to 10 mg/L of TCE in the vapor phase (Ensley, 1992). Effective long-term performance for more than 9 months in the laboratory, averaging more than 90% TCE removal, has been demonstrated (Guarini and Folsom, 1996). In these studies, G-4, and/or *Pseudomonas mendocina* were used along with phenol (for G-4) and/or toluene (for both G-4 and *P. mendocina*) as co-substrates for TCE degradation. The CSTR design and TCE treatment process has been effectively demonstrated in the field at Robins AFB, Georgia, and at F.E. Warren AFB, Wyoming using the 4,000 L pilot-scale bioreactor (Guarini and Folsom, 1996; Radian International, 1996). At F.E. Warren AFB, 85 to 90% total TCE removal was demonstrated over a 70-day period (Radian International, 1996).

Because of frequent SVE system shutdowns and poor TCE degradation in the CSTR during the McClellan AFB demonstration, the G-4 bacteria was re-inoculated into the CSTR on

two separate occasions. The re-inoculations were performed by completely draining the CSTR and then adding fresh water, fresh nutrients, and fresh G-4 bacteria. After the inoculation, the CSTR was operated in batch mode for approximately 10 to 14 days before continuous operations were started. During the second re-inoculation, the fresh water added to the reactor was passed through a granular activated carbon (GAC) filter and a cation exchange unit to remove impurities from the source potable water, before being added to the CSTR.

4.2 Treatment System Installation and Operation

This section discusses the logistics of the installation of the biotreatment system and associated equipment for the demonstration. The equipment consisted of a two-stage skid-mounted biotreatment unit (including a biofilter and a CSTR); an off-skid, 55-gal caustic drum (with a secondary containment) equipped with a pump; an off-skid, 55-gal phenol drum (with a secondary containment) equipped with a pump, and two storage tanks. The skid mounted units and associated equipment were positioned on a concrete pad directly adjacent to the SVE system at OU D, Site S. A field trailer, provided by the Air Force, was used to house the analytical instruments for on-site testing, including the on-site GC. This trailer was located at the field site, adjacent to the skid-mounted reactors, and was equipped with telephone and fax communications to facilitate daily communication between on-site and off- site personnel. The components of the system, and the utility, instrumentation, and control requirements are described in the following paragraphs.

A process and instrumentation (P&I) diagram for the two-stage biotreatment system is shown in Figure 4-1. The system consisted of a biofilter followed in series by a CSTR. As stated previously, phenol was used as the cosubstrate for cometabolic CAC degradation. The biotreatment system skid(s), 1,200-gal holding tanks, 55-gal containment vessels, and analytical supplies were shipped to the site and installed near the SVE system at McClellan AFB. A 55-gal drum of phenol, a 55-gal drum of caustic, the analytical reagents, and the biofilter medium also were shipped to the site.

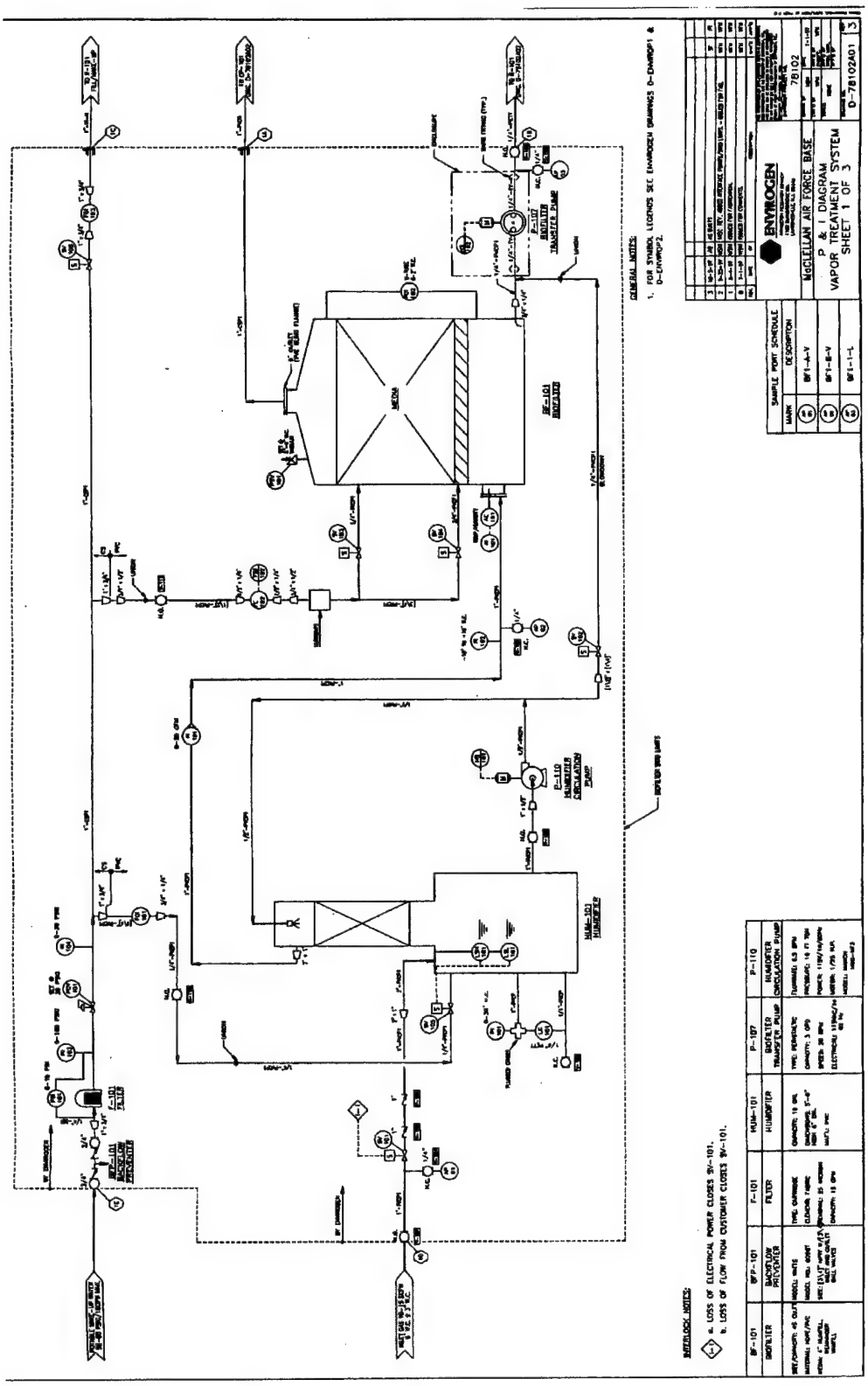

Figure 4-1. Process and Instrumentation Diagram (page 1 of 3)

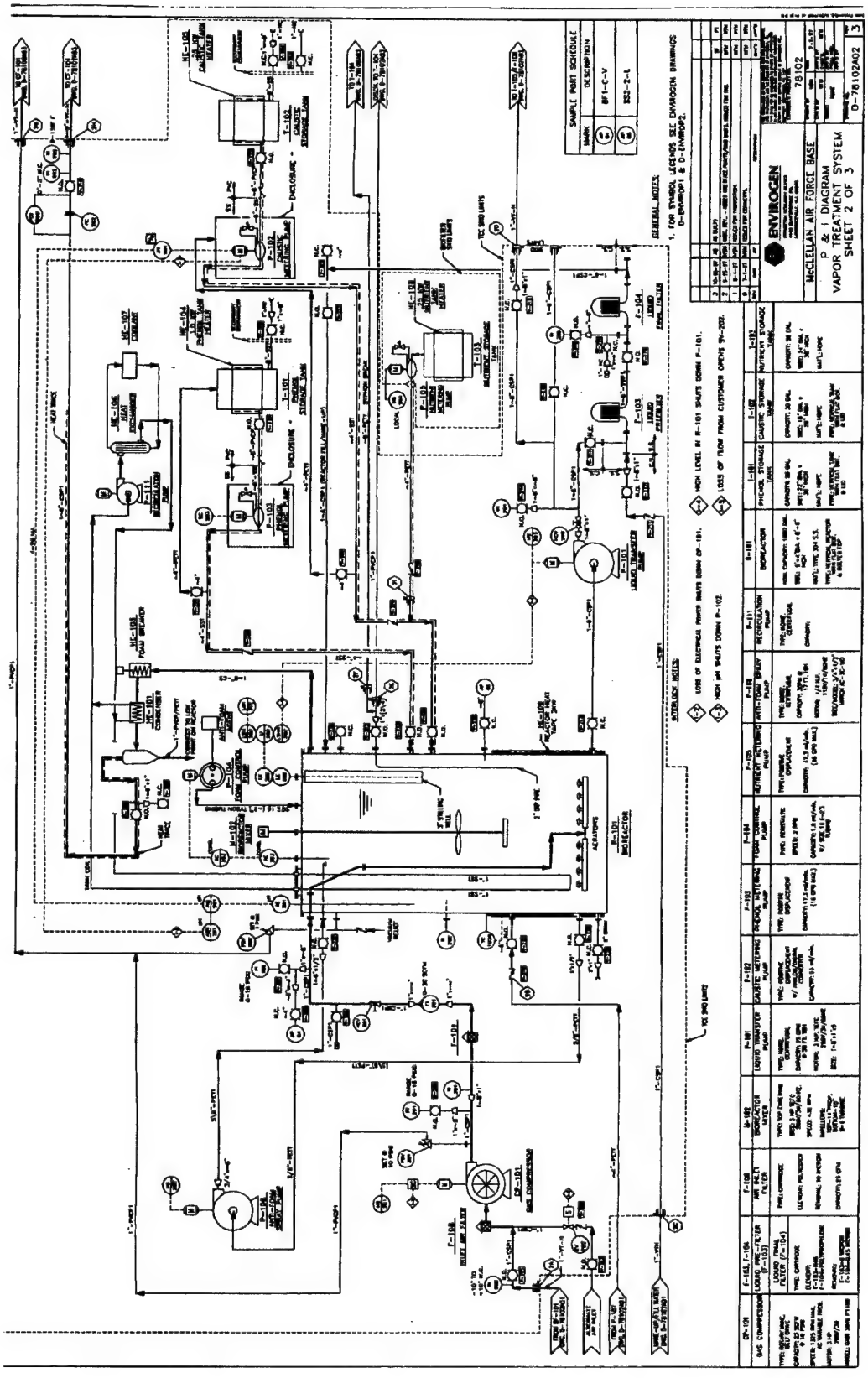

Figure 4-1. Process and Instrumentation Diagram (page 2 of 3)

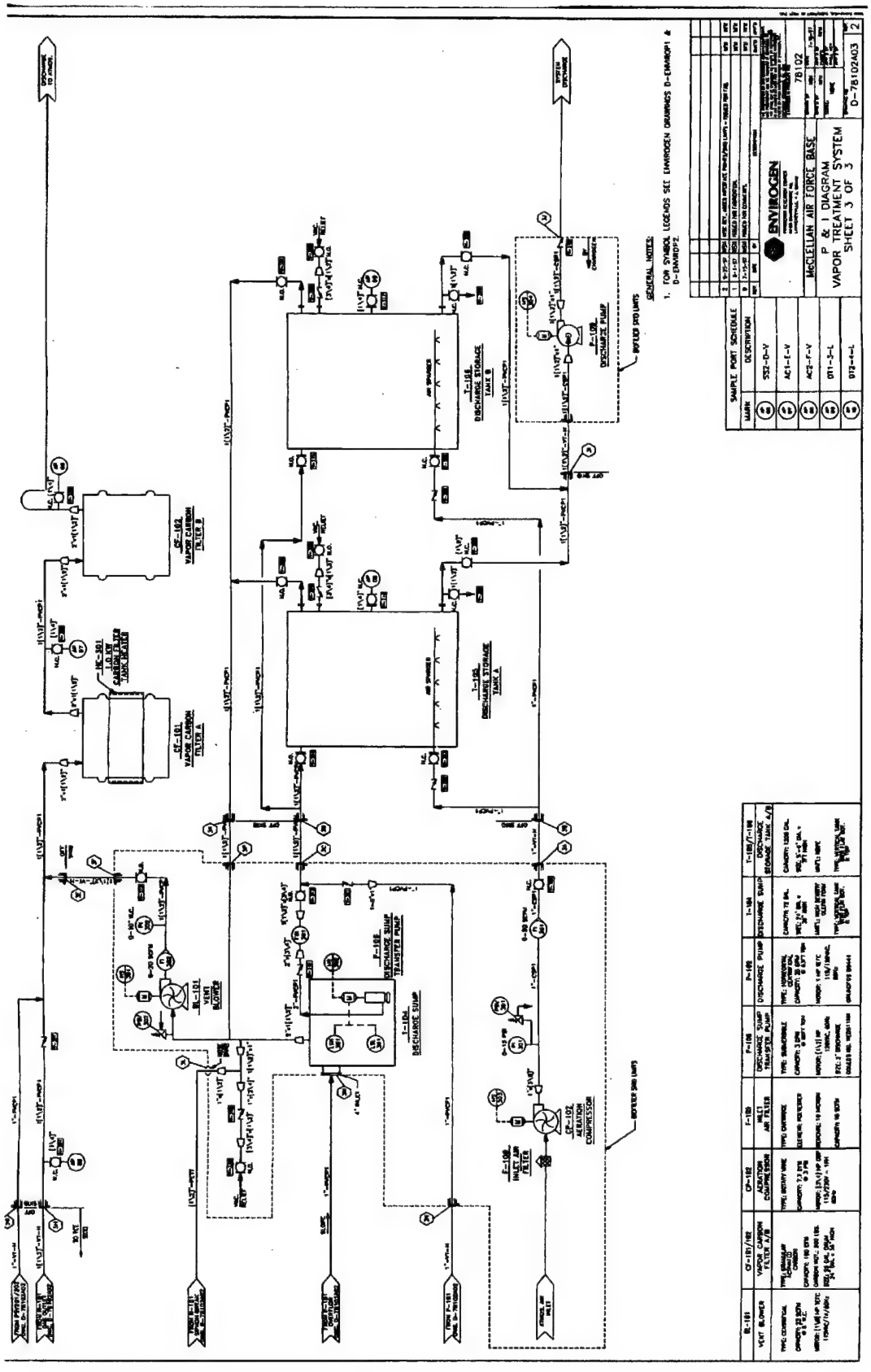

Figure 4-1. Process and Instrumentation Diagram (page 3 of 3)

After the reactors were delivered to the site, the system was assembled and power was supplied via on-site transformers. All on-line monitoring instruments (such as pH probes and vapor flowmeters) were checked and calibrated according to the manufacturer's instructions. All pressurized lines, connections, and fittings were checked for leaks using a soapy water solution. Loose fittings were adjusted to eliminate leaks.

The final hookup to the process gas stream was not connected until the operation of the system had been tested with ambient air. After the power was connected and the system was taken through the shakedown procedures, the process gas influent line to the system (i.e., the SVE off-gas) was connected to the SVE off-gas manifold leading to the on site cat-ox system. The connection was made from the SVE blower into a blank pipe connection, provided by the Air Force. As previously described, the vapor effluent from the biological treatment system was discharged to the atmosphere after treatment through the two carbon canisters in series. During operation, the effluent from the first canister was monitored daily using a portable PID and the on-site GC to assess breakthrough of VOCs. Weekly checks were made, as needed, by sending grab samples to an off-site laboratory for VOC analysis. When the PID reading from the first canister equaled or exceeded 95% of the influent reading, fresh carbon was delivered to the site. The first canister was removed and the second canister was used as the first canister in the off-gas treatment series.

4.2.1 First-Stage Biofilter

The first-stage biofilter reactor had approximate dimensions of 4 ft × 4 ft × 5 ft (length, width, depth [LWD]). The biofilter unit was positioned on a skid, measuring approximately 10 ft × 5 ft. The skid also contained a small humidification chamber, biofilter transfer pump, nutrient feed tank, and sump pump discharge tank for the CSTR; air blower for the wastewater storage tanks; discharge pump for liquid discharge; and vent blower. Severe vacuum pressure on the biofilter reactor resulted in on-site personnel having to use reinforcement techniques to prevent the biofilter lid from collapsing. This problem was reduced when the biofilter medium was placed in the reactor, thereby minimizing the vacuum induced by the site conditions.

4.2.2 Second-Stage CSTR

The field-pilot CSTR skid-mounted system had approximate dimensions of 11 ft × 8 ft × 11 ft (LWD) and weighed approximately 10,000 lb. The cylindrical bioreactor vessel was approximately 6 ft in diameter and 10 ft tall, and was approximately 750 gal in volume. The system was operated in a stirred-tank mode using a mixer to facilitate mass transfer. The equipment required three-phase, 460-volt power rated at 70 amps. The vessel and piping was constructed mostly of stainless steel. The system pH was automatically controlled using a 25% caustic sodium hydroxide (NaOH) solution. The system had two on-skid chemical feed systems for the addition of phenol and the caustic NaOH. The reactor water temperature was controlled via an on-skid heater and refrigeration system. Nutrients were continually added to the stirred tank from the nutrient feed tank located on the biofilter skid. The nutrients added ranged from 135 to 450 g/day of NH_4NO_3, and 50 to 150 g/day of KH_2PO_4. About 5 to 10 g of trace elements were added per day. The system was capable of handling up to 20 cfm of air flow, but the flow generally was maintained at 5 to 10 scfm. The vapor effluent from the biofilter was piped directly to the CSTR system during continuous operation.

4.2.3 Utility Requirements

All utilities installed for this technology demonstration were nonpermanent. Table 4-1 lists the utility requirements for the skid-mounted biological treatment system, and the following paragraphs describe the hookup and utility connections and requirements for the two-stage field-pilot system.

Table 4-1. Summary of Utility Requirements

Utility	Requirement
Potable water	Instantaneous flow = 10 gpm @ 30 to 60 psi. Average daily water consumption up to 100 gpd.
Electricity	CSTR Skid: 460-VAC, 3-phase, 4-wire, 70-amp power. Biofilter Skid: 220-VAC, single-phase, 20 amp power. Additional power was required for the mobile laboratory trailer, and was supplied by the Air Force.
Liquid waste	Up to approximately 1,000 gallons per week.

VAC = Voltage of alternating current.

4.2.3.1 Reactor Makeup Water

Potable water, used for the makeup water, connected by a ¾-in. hose with maximum instantaneous flow equal to 16 gpm at 30 to 60 psi. Potable water was provided by the Air Force via an on-site source. Daily water consumption was in the range of 0 to 150 gpd. A backflow control device was installed to prevent the return flow of reactor water into the Base water system. The water service installation adhered to McClellan AFB standards for service connections. During the second reinoculation of the CSTR the potable water was treated with a GAC filter and a cation exchange unit to remove potential impurities in the water.

4.2.3.2 CSTR Liquid Discharge

The storage tanks were equipped with level-sensing devices that shut off the water supply solenoid valve and the biofilter transfer pump to prevent overflow in the storage tanks. The wastewater was stored in the 1,200-gal storage tank prior to disposal. Before being disposed, the wastewater was sampled and analyzed by the off-site laboratory to determine whether the VOC concentrations met on-site effluent discharge requirements.

4.2.3.3 Feed Gas and Treated Gas

McClellan AFB provided a 2-in. blank flange from the SVE system. The hookup/reduction from this valve to a 1.5-in. female national pipe thread (FNPT) connection was in turn connected to the flange for the gas feed. Feed gas piping was sized for a maximum pressure drop that would not exceed 2 in. of water. The biotreatment system was designed to accept the vapor discharge from the SVE system blower. A pressure-relief valve upstream of the biofilter was used to reduce the pressure from the SVE system to approximately 2 in. of water. The pressure-relief valve was designed to accept pressures up to 1 psi. Humidity in the gas stream was addressed by passing the process gas stream through a humidifier. If the humidity fell outside the desired range, a heat exchanger in the humidifier recirculation loop was used to return the humidity back to the desired range. Gas flow into and out of the pilot system was rated for up to 20 cfm; the average flowrate was 7 scfm. The vapor effluent from the biotreatment system was discharged through two 200-pound carbon canisters before being discharged to the atmosphere.

4.2.3.4 Electrical Power

The biofilter skid was designed for single-phase 220-VAC power rated at 20 amps. The CSTR skid was designed to accept a 460-VAC, 3-phase, 4-wire, 70-amp power supply. Electrical power for the system was supplied by the existing on-Base source, i.e., an electrical transformer that was conveniently located near to the site. Power was hooked up to each skid using dedicated feed connections on the main control panels. Electrical servicing was performed by a qualified, licensed electrician and was done in compliance with National Electrical Code (NEC) and any applicable local electrical codes. Electrical receptacles were installed with ground-fault circuit interrupters. The design complied with NEC specifications in the National Fire Protection Act (NFPA) 70 and all applicable McClellan AFB standards for electrical services.

4.2.3.5 Wastewater

Liquid discharge water from the biotreatment system was stored in 1,200-gal storage tank(s) prior to disposal.

4.2.3.6 Biofilter Media

The biofilter medium was shipped to the site in 55-gal drums. The medium was stored for less than 1 month before the biofilter was loaded. No secondary containment was required.

4.2.3.7 Process Chemicals

Requirements for the materials storage of the process chemicals that were used during the demonstration are described as follows:

- **Ammonium Chloride and Potassium Phosphate.** A 100-lb bag each of ammonium chloride and potassium phosphate was stored on-site in separate steel drums.

- **Sodium Hydroxide (NaOH).** A 25% (w/w) (weight by weight) sodium hydroxide solution was shipped to the site in a 55-gal drum that was stored on site in a steel secondary containment vessel. NaOH was drawn from the drum using a pump, with piping directed to the caustic feed tank on the CSTR skid.

- **Phenol.** 90% (w/w) phenol was shipped to the site in a 55-gal drum, which was stored on site in a steel secondary containment vessel. Material was

drawn from the drum using a drum pump, with piping directed to the phenol feed tank on the CSTR skid.

- **Ethanol.** Ethanol was shipped to the site in a 55-gal drum, which was stored on site in a steel secondary containment vessel. Material was discharged from the drum using a drum pump.

- **Compressed Gases (Hydrogen, Nitrogen, Air, and Calibration Gases).** Compressed gases for the analytical instruments (i.e., the on-site GC) were shipped to the site in standard gas cylinders. The cylinders were secured within the mobile laboratory trailer.

4.2.4 Material Storage

The following materials were used or generated during the technology demonstration, and required some form of materials storage: sodium hydroxide, phenol, ammonium chloride, potassium phosphate, compressed gases for the field GC (i.e., hydrogen, nitrogen, and air), calibration gases for instrumentation, and wastewater.

4.2.5 Well Installation, Drilling, and Sampling

Not applicable for this project.

4.3 The Eight Phases of the Technology Demonstration

Upon completion of the equipment installation, system operations began on November 3, 1997. The plan of operation included the following eight conceptual phases.

1. Utilities Connection and System Shakedown
2. Abiotic Testing
3. Startup and Acclimation
4. Testing, Operation, and Data Collection and Analysis
5. Maximization of Vapor Throughput and Contaminant DREs
6. pH-Attenuated Control
7. Performance Monitoring
8. Shutdown, Demobilization, and Site Restoration.

Only phases 1, 2, 3, 4, 7, and 8 were completed during the demonstration. Phases 5 and 6, which involved process optimization, were not conducted because of the inadequate performance of the CSTR.

4.3.1 Operating Phase 1: Utilities Connection and System Shakedown

Following the installation of the two skid-mounted units and associated piping and equipment, the utilities were connected and the system shakedown was performed. The biofilter skid required a single-phase 220-volt power source rated at 20 amps. During operation, the volumetric flowrate of the biofilter system discharge water was approximately 24 gpd, which was pumped directly to the CSTR. The maximum water consumption during the demonstration was less than 30 gpd.

The biofilter medium compartment was loaded on December 10, 1997 with approximately 45 ft^3 of biofilter medium, made up of composted bark nuggets, compost, and fertilizer. Once loaded, the biofilter medium rested on an inert layer of sorbent material called Humfil™ (U.S. Patent 5,445,660). The biofilter was equipped with sets of pressure-compensating irrigation hoses to provide even distribution of water across the medium bed and humidification layer. Both sets of hoses were operated on timed cycles to maintain 60 to 70% (w/w) medium moisture levels. One set of hoses was positioned at the top of the bed, approximately 6 inches below the medium surface. The other set was positioned above the Humfil™ layer, which acted as a final humidification zone for the influent vapor stream. In addition to providing moisture, the upper irrigation hoses were used to periodically provide inorganic soluble nutrients to the medium using slow-dissolving tablets.

Discharge water from the biofilter was added continually to the stirred-tank vessel using a peristaltic pump. Additional water was added to the vessel from a potable water supply at a rate that did not exceed 100 gpd throughout the demonstration. The wastewater effluent exited from the CSTR by passing over an overflow weir to a discharge sump. When the discharge sump filled, the sump pump transferred the wastewater to one of the two 1,200-gal polypropylene discharge storage tanks for treatment or disposal.

4.3.2 Operating Phase 2: Abiotic Testing

After the biotreatment system and analytical instrumentation were set up and checked, system operation was started. Before inoculating the biofilter or CSTR, both reactor stages were operated abiotically under the expected normal operating conditions. Vapor samples were analyzed daily, using with the on-site PID and GC, to assess abiotic system losses.

The purpose of initially operating the system abiotically in the absence of micro-organisms, medium, or nutrients was threefold:

1. To troubleshoot initial system operating problems without jeopardizing the integrity of the system.
2. To assess the magnitude of any contaminant losses from the system not attributed to biological degradation, such as adsorption, permeation, or gas leaks.
3. To ensure that losses under ideal operating conditions were minimized.

Operation did not begin until an acceptable mass balance was attained (i.e., greater than 90% overall recovery from the inlet of the biofilter to the outlet of the CSTR). During the entire abiotic control period, the operating parameters of the system were monitored daily.

After troubleshooting the system, operating data was collected for 72 hours. Once steady state was achieved, influent and effluent vapor grab samples and effluent discharge liquid samples were collected and sent to off-site laboratories. (This sampling event occurred on December 3, 1997.) The vapor samples were analyzed using U.S. EPA TO-14 analyses. Using the on-line and grab sample data, overall and compound-specific mass balances were calculated to assess abiotic losses from the system in the absence of biological activity.

4.3.3 Operating Phase 3: Startup and Acclimation

Following the abiotic control phase of the demonstration, the first-stage biofilter was loaded with biofilter packing and inoculated with Phenobac® (Polybac Corporation, Bethlehem, Pennsylvania). Phenobac® is a general, nonspecific, mixed inoculum previously used by Envirogen for biofilter applications. Samples of the packing material were collected shortly after loading the system for total organic carbon (TOC) and lipid (Microbial Insights, Inc.; Knoxville,

Tennessee) analyses. At the end of the test, samples were collected again and analyzed for TOC and lipids to assess the extent of microbial growth in the biofilter medium.

The inoculum, G-4, was shipped overnight on ice and was used to inoculate the CSTR. After inoculation, the second stage was operated for 1 week with cosubstrate feed, but without contaminant feed, to build up biomass in the reactor until a biomass protein concentration of 1 g/L was established, as measured using a protein assay kit (BCA protein assay; Pierce, Rockford, Illinois). The phenol concentration in the bioreactor was monitored routinely using the wet-chemical analysis method. Ambient (noncontaminated) air was supplied to the system during this period via a bypass air intake line. It took 10 days to grow the biomass to the 1 g/L density; during a second inoculation event, the desired biomass levels were reached in approximately 8 days.

After the biomass was grown, contaminated vapors from the SVE system were supplied to the reactors at a target flowrate of 7 scfm. The cosubstrate addition rate was adjusted to maintain a cosubstrate to CAC feeding ratio of 12:1, based on the expected influent CAC concentrations. Before supplying the contaminated off-gas to the reactors, a phenol-degradation assay was conducted to assess the health of the microbial culture. The acceptable phenol degradation rate was in the range of 90 to 100 nmoles phenol/mg protein/min. Vapor-phase contaminant concentrations were analyzed daily using the PID and the on-site GC. The sample collection points for the PID and on-site GC monitoring were (1) the influent to the system; (2) the effluent from the biofilter unit; (3) the system effluent; and (4) the effluent of the GAC polishing unit for compliance sampling. After approximately 1 week, the system was assumed to be acclimated to the SVE feed stream, and process testing was begun on December 17, 1997. Grab samples for VOC analyses also were taken and analyzed off-site.

Several losses of biomass in the CSTR occurred during the demonstration. These losses occurred during the SVE system shutdowns (see Table 5-16). The precise reason for the biomass losses is unknown; most likely, they were due to washout or endogenous decay. When these losses affected system performance, the process gas and potable water flows to the system were discontinued and the system was fed phenol only in batch mode to increase the G-4 biomass to acceptable levels; ambient air was fed to the CSTR during these periods to maintain aerobic conditions.

4.3.4 Operating Phase 4: Testing, Operation, and Data Collection and Analysis

For the first 2 months of operation, the cosubstrate feeding rate was adjusted, as needed, to increase system performance. The vapor flowrate through the system was maintained at approximately 7 scfm. Initial cosubstrate (phenol) feeding rates were approximately 2,000 g/day. The phenol was supplied to the reactor as a 10% (w/w) solution. The CSTR steady-state phenol concentration was less than 0.5 mg/L, and the phenol addition rate was adjusted accordingly to maintain this concentration. The phenol concentration in the CSTR was measured periodically during field operations to adjust the phenol addition rate accordingly.

Sample ports were located at key points throughout the system (see Figure 4-1). These included vapor sampling ports for the SVE off-gas influent to the biofilter, the influent to the CSTR, and the effluent from the CSTR, and sample ports between the two carbon canisters and the effluent from the second carbon canister. Also, a liquid sampling port was located on the effluent line from the CSTR.

Gas-phase VOC concentrations were measured from grab samples collected during reactor operation. A Hewlett Packard (HP) 5890 GC equipped with a 60-m SPB-1 wide-bore capillary column attached to a flame ionization detector (FID) was used to separate and quantify the VOCs. Samples were collected in pre-cleaned Tedlar™ bags. A 10-mL sample was pulled from the bag with a glass syringe fitted with a Teflon™ plunger. The gas sample was injected by hand into the heated six-port injection valve fitted with a 2-mL loop. The area counts for the compounds were stored as computerized files using the GC's analytical station data acquisition software. The VOC concentrations were calculated by using the response factors generated from a multi-point calibration from injections of known standards.

A field GC equipped with a FID was used to monitor specific VOCs in the grab samples collected during reactor operation. The GC was calibrated prior to use using a known calibration standard gas mixture supplied by Praxair, Inc. The certified gas standard contained 10.1 ppmv of 1,1-DCA, 14.9 ppmv of toluene, 19.8 ppmv of 1,2- dichlorobenzene (DCB), 40.1 ppmv of PCE, 39.9 ppmv of TCE, 39.7 ppmv of 1,1,1-TCA, and 10.1 ppmv of 1,2-DCE with a balance gas of nitrogen. A separate Scotty II bottle of VC, at 9.99 ppmv, also was used to calibrate the instrument for VC. Secondary standards of progressively lower concentrations that provided five-point concentration standards were prepared from the primary gas standard.

A check standard of known concentration was analyzed daily using the calibration gas mixture on site. If the measurement fell outside ±20% of the known check standard concentration, then the check standard was rerun. If the measurement continued to fall outside ±20% of the known concentration, the GC was recalibrated.

The instrumentation and controls for the biotreatment system consisted of manual components. Automatic shutdown controls were used for critical system components (i.e., blowers and pumps). Flow control from the SVE system was adjusted manually using valves.

Automatic control was provided to protect equipment and personnel in the event of equipment failure. The first level of protection was a local emergency shutdown switch of all pilot equipment. Automatic control also interlocked the pilot system blower(s) with the SVE system blower(s). The pilot system blower(s) would not operate unless the SVE system was in operation.

A backflow preventer was installed upstream of the pilot treatment system to prevent backflow of process air into the SVE off-gas manifold.

The reactors were designed for outdoor use in nonexplosive environments. The blower was set to automatically shut down in the event of one of the following conditions: (1) high blower differential pressure, (2) high blower discharge temperature, or (3) high liquid temperature. During the demonstration none of these conditions occurred.

To meet the project objectives the operating parameters of the system were monitored daily for on-site analyses, and liquid and vapor samples (both on-line and grab samples) were taken approximately weekly for off-site analysis. Steady state was indicated by stable contaminant concentrations in the effluent vapor from both the biofilter and the CSTR. The on-site GC and PID readings were used to assess steady-state conditions, and off-site analyses were conducted to establish system performance.

During the demonstration, minor upsets occurred resulting in the discharge of residual phenol with the wastewater. To minimize the amount of phenol in the residual wastewater, the discharge tanks were sparged with ambient air to promote phenol degradation in the waste tanks. A blower was used to vent vapors from the discharge sump and wastewater storage tanks to the activated carbon. The vapor effluent from the CSTR was processed through the same carbon canisters before being discharged to the atmosphere.

Mass balances were calculated by measuring VOC concentrations in the influent and effluent gas streams and in the effluent liquid waste stream using total and soluble aqueous-phase VOC analyses.

4.3.5 Operating Phase 5: Maximization of Vapor Throughput and Contaminant DREs

Because of unsatisfactory system performance, the process could not be optimized. Throughout the study, every possible effort within the project's resources was made to achieve DREs approaching 95%.

TCE was the primary target contaminant, followed by 1,2-*cis*-DCE and VC. Removal of toluene in the biofilter was monitored, but was not optimized. Because the 95% target DRE was not achieved by the time the optimization period began, the goal of the optimization period was to try and achieve 95% DREs of all contaminants. The optimization period was expected to last approximately 1 month.

As with most experimental biological processes, the vapor throughput adjustments required careful judgement of the on-site personnel involved in the demonstration. The process gas flowrate initially was set to 7 scfm and was kept relatively constant throughout the testing period. Because 95% DREs were not achieved at any of the desired flowrate settings, the off-gas flowrate was maintained at approximately 7 scfm. System flow was never decreased below 5 scfm.

During this period, the operating parameters of the system were monitored daily, and liquid and vapor samples (both on-site and off-site laboratory grab samples) were taken for analyses according to the sampling schedule. Data were analyzed to meet the sampling and QA/QC objectives of the demonstration.

4.3.6 Operating Phase 6: pH-Attenuated Control

Because the target DREs were never achieved, the experiments that were to examine the effects of pH on the re-circulating water within the CSTR to reduce the activity of the biomass within the system were not performed.

4.3.7 Operating Phase 7: Performance Monitoring

Performance of the biotreatment system was evaluated on the basis of data generated during the demonstration. Evaluation of performance relied on the collection of sufficient data during the demonstration. Significant process parameters that were monitored during operation are presented in Table 4-2, along with the respective methods used.

Table 4-2. Process Parameter Monitoring Methods

Process Parameter	Monitoring Method[a]
Influent vapor concentration	Tedlar™ bag grab samples for portable PID analysis and on-site GC (U.S. EPA Methods 8010/8020), and evacuated canister grab samples for individual VOCs (TO-14)
Biofilter (Stage 1) Effluent vapor concentration	Tedlar™ bag grab samples for portable PID analysis and on-site GC (U.S. EPA Methods 8010/8020), and evacuated canister grab samples for individual VOCs (TO-14) and for total non-methane hydrocarbons (TO-12)
CSTR (Stage 2) Effluent vapor concentration	Tedlar™ bag grab samples for portable PID analysis and on-site GC (U.S. EPA Methods 8010/8020), and evacuated canister grab samples for individual VOCs (TO-14) and for total non-methane hydrocarbons (TO-12)
Vapor flowrate	Orifice plate, rotameter
SVE feed gas pressure	Pressure gauges
Liquid discharge concentration	(VOCs) U.S. EPA Method 8620/624, (BODs) U.S. EPA Method 405.1, (TSS) U.S. EPA Method 160.2, total protein, optical density
Liquid discharge rate	Flow totalizer, water levels in the discharge storage tanks
CSTR make-up water usage rate	Flow totalizer
Biofilter water usage rate	Flow totalizer
Humidifier water usage rate	Flow totalizer
Biofilter packing pressure drop	Pressure gauges above and below the packing
Nutrient addition rate	Liquid level in nutrient addition tank(s)
Nutrient concentrations in CSTR (Stage 2)	HACH® test kits
Cosubstrate addition rate	Liquid level in the cosubstrate addition tank over time
Caustic addition rate	Liquid level in caustic addition tank
Electrical power consumption	An electrical meter was used to total the power consumption of the system
Biofilter packing	(TOC) U.S. EPA Method 415.1, lipid analysis (Microbial Insights, Inc., Knoxville, Tennessee), Toxicity Characteristic Leaching Procedure (TCLP)

(a) Refer to Appendices E and F for the detection limits (DLs) and practical quantitation limits (PQLs) for the vapor and liquid analyses.

4.3.8 Operating Phase 8: Shutdown, Demobilization, and Site Restoration

After all data required to assess the effectiveness of the technology were obtained, the system was shut down on April 15, 1998. All final operating conditions and field measurements were documented. A final biofilter packing sample was taken. The biofilter packing was a composite sample taken from three different depths. The biofilter composite sample was analyzed for TOC and lipids, to assess the extent of biological growth. In addition, biofilter packing samples were analyzed for TCLP parameters.

Fresh air was processed through the CSTR for 24 hours to strip any remaining VOCs from the water. GAC polishing was maintained on line during all decontamination activities. The liquid was discharged to the 1,200-gal holding tanks for analysis before disposal. All water tested (SW8260 analyses) was found to be acceptable for treatment by the base wastewater treatment operations. The CSTR was decontaminated by running clean water and fresh air through the system. The biofilter packing material was removed from the biofilter vessel and packaged into 55-gal drums for disposal in a hazardous waste landfill, as necessary. The spent carbon, phenol, and caustic canisters were transported to a hazardous waste disposal facility.

The biofilter, CSTR skids, and 1,200-gal tanks were secured and shipped back to Envirogen, along with other equipment at the site; any rented equipment was returned to the suppliers. After all the equipment was removed, the site was returned to its original condition, and an inspection was performed by a McClellan AFB representative. All temporary connections that were installed for the demonstration to the existing SVE system were removed, and the site was vacated.

4.4 Sampling Strategy and QA/QC Results

A comprehensive and accurate performance evaluation of the biofilter system depended on obtaining a complete, representative, and consistent data set chronicling the results of the demonstration. The data characterized the original contaminant concentrations and the amounts and rates of the contaminant removal efficiency of the two-stage reactor system. The project sampling plan in this section specifies the sampling locations, frequency, methods, chemical analyses, and administrative procedures that were used during the demonstration.

The biotreatment demonstration included a field sampling protocol that was separated into two components: (1)technology performance sampling, and (2)quality assurance sampling. Samples were collected using the methods for gathering, numbering, documenting, handling, analyzing, and decontaminating described in this report.

4.4.1 Pre-Demonstration Sampling

No pre-demonstration sampling was performed for this application. Data on contaminant concentrations from previous site characterization activities were used to determine reactor parameters and experimental conditions (Battelle and Envirogen, 1997a).

4.4.2 Technology Operation

The biofilter matrices sampled included SVE off-gas influent to the biofilter, biofilter effluent vapor from Stage 1 of the biofilter, biofilter effluent vapor from Stage 2 of the biofilter, and biofilter recirculation water in Stages 1 and 2 of the biofilter. The number of samples collected were based on the duration of operation (approximately 12 weeks), and the anticipated time scale for reaching equilibrium. However, frequent shutdowns of the SVE system and other operational limitations restricted the number of possible representative samples. In total, six sets of off-site samples were collected during the demonstration, including the abiotic and startup samples. The primary objective of the demonstration was to sample frequently enough to define the removal rates achieved by the biofilter system during each phase of operation, and to perform mass balance calculations on the biofilter unit. The mass balances were used to ensure that mass was conserved in the biofilter, and to help characterize partitioning of the various contaminants within the reactor.

4.4.2.1 Process Sampling

During each workweek, influent and effluent vapor concentrations were monitored daily, using a hand-held PID and grab samples injected into the on-site GC to provide gas-phase VOC concentrations. Limited Base access prevented weekend sampling. Vapor grab sampling ports were located at (1) the influent to the system; (2) the effluent from the first stage; and (3) the effluent from the second stage. The field GC was calibrated daily against standard gas mixtures

with known concentrations of seven representative VOCs, including PCE, TCE, 1,1-DCA, 1,2-*cis*-DCE, VC, 1,1,1-TCA, and toluene.

Six times during the field demonstration, liquid samples from each reactor stage and vapor samples from the reactor gas sampling ports were collected simultaneously for use in the mass balance calculations. The liquid-phase samples were analyzed for aqueous-phase VOCs, TOC, and chloride. For the mass balance tests, vapor-phase samples were analyzed for vapor-phase VOCs using U.S. EPA Methods TO-12 and TO-14.

All the aqueous- and vapor-phase samples were analyzed on-site or by the outside laboratories contracted by Battelle in accordance with the methods shown in Table 4-3. On-site vapor samples were collected in precleaned and pretested Tedlar™ bags. Tedlar™ bags were precleaned by filling and purging the bag with compressed air at least three times. Background vapors were measured using the hand-held PID for PID analyses and using the on-site GC for GC analyses. Summa canisters were precleaned by the contract laboratory and were tested on-site using a pressure gauge to ensure vacuum pressures in the canisters.

Liquid samples were drawn in precleaned 40-mL volatile organic analysis (VOA) glass containers. Prior to sampling, the sample ports were flushed with the process water by opening the valves and allowing approximately 100-mL of process water to flow into the on-site liquid effluent containers.

Table 4-3 shows the analytical methods used during this demonstration, and specifically lists the analytical method, sample container type and size, preservative, and holding time required for each type of analyte.

4.4.2.2 System Performance Under Stressed Conditions

Because the predetermined DRE of 95% was not met during steady-state operation, stress tests were not conducted.

4.4.2.3 Maximization of DREs and System Throughput

Through the duration of the field demonstration, attempts were made to achieve the 95% DRE objective. A secondary goal for the duration of the project was to meet the sampling and QA/QC objectives. Section 4.4.5 provides additional information on sampling and QA/QC objectives.

Table 4-3. Analytical Methods and Requirements

Sample Matrix	Analyte Type	On or Off Site	Method Name	Container Type	Container Size	Preservative	Holding Time
Process Water	VOCs	Off	SW 846 8240	VOA Vial	40 mL	Zero headspace HCl: pH<2 Cool: < 4°C	14 days
	Phenol	On	4-amino-antipyrine	Glass	40 mL	None	Analyze immediately
		Off	4-amino-antipyrine	VOA vial	40 mL	Zero headspace HCl: pH<2 Cool: < 4°C	14 days
	pH	On	U.S. EPA 150.1	Glass	Beaker	None	Analyze immediately
	NH_4-N	On	U.S. EPA 8038	Glass	Beaker	None	Analyze immediately
	PO_4-P	On	U.S. EPA 8048	Glass	Beaker	None	Analyze immediately
	Chloride	On	Chloride electrode	Glass	Beaker	None	Analyze immediately
	Protein	On	BCA protein Assay Kit[a]	Glass	Beaker	None	Analyze immediately
	BOD_5	Off	U.S. EPA 405.1	Glass	500 mL	Zero headspace Cool: < 4°C	Analyze immediately
	TSS/VSS	Off	U.S. EPA 160.2	Glass	100 mL	Zero headspace Cool: < 4°C	Analyze immediately
Vapor	VOCs	On	SOP: McAFB-027	Stainless steel	Tedlar™ bags	Keep Dark	Analyze immediately
		Off	U.S. EPA TO-14	Stainless steel	Summa	None	14 days
	TNMOC	Off	U.S. EPA TO-12	Stainless steel	Summa	None	14 days
Biofilter Compost	Lipids	Off	PLFA[b]	Polyethylene	500 mL	Cool: < 4°C	14 days
	TOC	Off	EPQ 415.1	Polyethylene	500 mL	Cool: < 4°C	14 days
	TCLP	Off	U.S. EPA 1311	Glass	500 mL	Cool: < 4°C	14 days

(a) Pierce (Rockford, Illinois); Kit 23225.
(b) Microbial Insights (Knoxville, Tennessee).
BCA = bicinchoninic acid.
BOD_5 = 5-day biological oxygen demand.
HCl = Hydrochloric acid.
NA = Not applicable.
NH_4-N = Ammonia – nitrogen.
PLFA = Phospholipid fatty acid.
PO_4-P = Phosphate – phosphorus.
SOP = Standard operating procedure.
TNMOC = Total nonmethane organic carbon.
VOA = Volatile organic analysis.
VSS = Volatile suspended solids.

4.4.3 Post-Demonstration Sampling

Post-demonstration characterization sampling usually is conducted to characterize the test site upon completion of the system demonstration. Post-demonstration characterization was not required for this demonstration because it was being performed on an existing process stream.

4.4.4 Shut-Down Monitoring

Not applicable for this project.

4.4.5 Quality Assurance Sampling

The purpose of the field QA/QC program was to provide a measure of data quality. The program involved the collection of field duplicates, equipment blanks, liquid and vapor blanks, and field blanks. QA samples collected during the demonstration are described below.

For the on-site vapor analyses, check standards with known concentrations were analyzed daily using a calibration gas mixture on site (McClellan AFB SOP:McAFB-027). The GC system was calibrated if duplicate check standard measurements fell outside ±20% of the known check standard concentrations. In addition, influent, effluent, and intermediate reactor vapor samples were collected biweekly using evacuated canisters for U.S. EPA TO-12 and TO-14 analyses by Air Toxics, Ltd., an independent outside California certified laboratory. These results were used to quantify system performance, mass balances, and DREs. As a QA/QC check of the chloride measurements made using the chloride probe, the measurements from the probe were compared at four time points with ion chromatography measurements performed using U.S. EPA Method 300 by an outside laboratory. Data review procedures were in accordance with SOP McAFB-028, as noted below. Also data validation was in accordance with SOP McAFB-029, as noted below.

4.4.5.1 Duplicate Sample Collection

Duplicate samples were collected to aid in the evaluation of the precision of the overall sampling and analysis event. Duplicates were collected as replicate samples that represented the primary sample. Duplicate samples were conducted at a minimum 5% frequency (one of every 20 samples). Methods for collection of the field duplicates are described in following subsections.

4.4.5.2 Biofilter Process Water

Primary and duplicate sample containers were filled completely from a single grab. Duplicate samples were collected sequentially from the same source or sampling device after sample ports were flushed with process water.

4.4.5.3 Vapor

Primary and duplicate canisters were filled concurrently from adjacent sampling ports. The sampling time and date was noted in the field logbook. The field engineer ensured that adequate time was available during sampling to fill both canisters. This time was recorded in the field logbook.

4.4.5.4 Off-Site Laboratory Blanks

Blanks were collected in association with liquid and air samples at a 5% frequency. The blanks were analyzed by the same method(s) as the primary samples.

4.4.5.5 Liquid Blanks

Liquid blanks were prepared using organic-free water available on site or shipped from Battelle to the site. The organic-free water rinsate was collected directly into the appropriate sample containers. Sampling techniques used were noted in the field logbook. Blanks were collected in association with liquid samples at a 5% frequency.

4.4.5.6 Vapor Blanks

Air blanks were prepared using ultrapure, moisturized nitrogen at 5% frequency. Nitrogen gas was supplied to the site from an outside vendor. The nitrogen was delivered to the sample canister through a standard probe and flow-controller assembly to simulate actual field equipment conditions. The nitrogen stream was moisturized using an in-line impinger filled with organic-free water. Moisturizing of the nitrogen introduced to the blank sampling assembly was performed by passing/sparging the nitrogen from a pressurized cylinder through organic-free water. The moistened stream was then directed to the sample container.

4.4.5.7 Field Blanks

Field blanks were collected at a 5% frequency for liquid and vapor samples. The blanks were collected at a designated sampling location to simulate ambient sampling conditions. At the selected sampling locations, liquid or gas samples were collected in their appropriate sample containers and subsequent analysis of each field blank corresponded to that of the respective sample. Liquid field blanks were collected into VOA vials and gas samples were collected in evacuated Summa canisters.

4.4.6 Sampling Equipment and Field Procedures

This section describes the equipment and procedures that were used to collect samples for the analyses presented. The equipment used for these procedures was decontaminated according to the protocol outlined in Section 4.4.6.1.

Liquid samples were collected in specified containers. If required, samples were preserved with HCl to lower the pH to below 2.0 to stop all biological activity. The preservative was added to the vials before sampling. The pH was checked in a sample vial not used for analysis to ensure that the pH had dropped to below 2.0. Liquid samples were collected directly from the grab sample ports, which were equipped with 1/8-in.-diameter Teflon™ tubing. The ports and tubing were flushed with 100- to 500-mL of reactor process water immediately prior to sampling. The process water used for flushing was discarded with the process wastewater. The samples were poured slowly down the vial sidewalls, and the vials were filled to capacity with no headspace, as required.

Liquid samples for on-site analysis of pH, conductivity, and chloride concentration were collected in the manner described above. Liquid temperature was monitored directly in the reactor using a temperature probe, thermocouple, and meter. Field analyses were conducted immediately after sampling.

Off-site process gas samples were collected in evacuated Summa canisters provided by the off-site laboratory in accordance with U.S. EPA Methods TO-14 or TO-12. The canisters were connected directly to the respective gas grab sample ports. Summa canisters were equipped with influent flow regulators to control the rate of sample collection. Negative pressures (vacuum) were checked using a pressure gauge provided by the laboratory before and after

sampling to ensure satisfactory sample collection. Gas samples were collected over a period of 30 to 60 minutes per sample to obtain 30- to 60-minute composite samples. The ports were designed such that duplicate gas samples could be withdrawn simultaneously.

On-site gas samples were collected in Tedlar™ bags that were precleaned by repeatedly evacuating them with compressed air. Before drawing any samples, the bags were tested with the on-site GC for the presence of residual contaminants, depending on the intended analysis. If residual contaminants were detected, the purging procedure was repeated, until background contaminants were nondetectable. Gas samples were hand-injected into the GC using glass syringes with Teflon™ plungers. The results of the analyses were recorded into the GC analytical station's computer; a hard copy is provided in Appendix D.

4.4.6.1 Decontamination Procedures

Contamination was associated principally with the constituents in the SVE system off-gas. Water cleaning to remove soil and contaminants was the primary feature of the equipment decontamination process. Two levels of equipment decontamination were implemented in two processes:

1. Level 1 involved the decontamination of the bioreactor unit and all components that came in contact with the contaminated off-gas.

2. Level 2 was a general decontamination process that applied to all on-site equipment used for sampling liquid and gas, including glassware, tools, or other equipment that directly contacted the sampled media.

The two decontamination processes are shown in Table 4-4. All biofilter equipment (Level 1) was decontaminated before entering and leaving the site. Level 2 was applied to decontaminate equipment between each use. Sampling equipment was put in a plastic-lined decontamination area after each sampling event.

Table 4-4. Decontamination Processes

Decontamination Level	Process
Level 1	Removed all loose dust
	Thoroughly cleaned with water
	Rinsed at lest three times with water
	Collected rinsate for proper disposal, as necessary
	Wore proper personal protective equipment (PPE) during decontamination process
Level 2	Removed all loose dust
	Thoroughly cleaned with potable water
	Scrubbed with Alconox™ or Liquinox™ and water
	Rinsed with distilled water
	Rinsed with methanol
	Rinsed with deionized/distilled water
	Air dried
	Collected rinsate for proper disposal, as necessary

4.4.6.2 Sample Designation

A sample identification system was used to identify each sample submitted for analysis. The purpose of the sample identification system was to ensure that each sample was given a unique sampling identifier, and to provide a suitable tracking system for all samples. This system helped prevent data problems such as lost data, conflicting results, and untraceable samples.

A listing of sample identification numbers was maintained in the field logbook and a separate sample record book. The field logbook was used for keeping general field notes. The sample record book was used for recording sample numbers and dates and to aid field personnel in tracking sample sequences. Additionally, individual sample numbers and collection dates and times were entered on a chain-of-custody record, which accompanied the samples en route to the laboratory.

4.4.6.3 Sample Handling, Preservation, and Shipment

Sample integrity was ensured primarily through proper handling, preservation, and shipment procedures. Table 4-3 specifies the containers, preservatives, and holding times for all analytical methods that were used during the demonstration.

All collected samples were properly labeled according to procedure and stored. The sampler secured the lid, cap, or port on the container and placed the sample in the appropriate storage or shipping container. Proper sample handling minimized the chance of sample contamination or loss. The following are the separate handling procedures used for liquid and vapor samples:

- **Liquid Samples**. Liquid samples for off-site analyses were collected in containers supplied by the analytical laboratory. Sample containers were shipped to and from the laboratory inside a cleaned cooler. Glass sample containers were packed in shock-absorbent packing material.

- **Vapor Samples**. Vapor samples were collected in evacuated Summa canisters supplied by the analytical laboratory or in precleaned Tedlar™ bags for injection into the on-site GC. Evacuated Summa canisters were packed in shock-resistant material for shipment to the analytical laboratory.

4.4.6.4 Sample Shipment

Samples were packed in appropriate shipping containers with packing materials that minimized the chance for breakage. All samples designated for off-site analysis were shipped to the laboratory via same- or next-day delivery, or were dropped off to the laboratory by on-site personnel. All applicable sample packaging and labeling requirements for interstate transport of hazardous materials were followed as defined in the Code of Federal Regulations (CFR) in 40 CFR 49, Chapter 1, Part 171. A chain-of-custody record accompanied each sample shipment. Samples collected for on-site analysis were carried to the on-site laboratory trailer and analyzed.

Upon receipt of each sample shipment by the off-site analytical laboratory, the coolers or shipment containers were inspected. No errors or problems were noted on the chain-of-custody record or reported to the staff responsible for the QA/QC of the demonstration.

4.4.6.5 Sample Holding Times

Sample holding times listed in Table 4-3 were followed to maintain sample integrity. Samples were collected and prepared for shipment on the same day. Samples that were not shipped on the day of collection were sent no later than the following day. If the contract laboratory was unable to process the samples on weekends, samples were not collected on

Fridays or Saturdays for off-site analysis. Prompt shipment ensured that the laboratory was given adequate time to perform analyses before expiration of holding times. Each sample that was collected during the demonstration was analyzed prior to the expiration of the sample holding time.

4.4.6.6 Sample Documentation

A sample documentation program ensured that all samples were properly tracked. The components of the sample documentation program included sample labels, custody seals, field logbooks, photographs, chain-of-custody forms, and laboratory logbooks. Each component of the sampling documentation was important in assuring that QA/QC objectives for the demonstration were met. Each component is briefly described below.

Sample Labels. Each sample collected was labeled with the sample designation number, sample type, date, and sampler's name. These data were entered in the appropriate spaces on laboratory-supplied labels. Entries were made in black, or blue ink at the time of sample collection. The completed sample label was affixed to the appropriate container immediately upon collection of the sample.

Custody Seals. Custody seals ensured that the stored samples had not been tampered with or otherwise exposed to potential contamination. Custody seals were used when containerized samples were shipped or were otherwise not in the immediate view of the collector. The collector signed two custody seals and placed one across the lid attachment point on each side of the cooler.

Field Logbook. On site personnel maintained a sampling field logbook for all sampling events. The field logbook was a bound notebook with numbered pages. All entries were made in black or blue ink. The sampler kept custody of the sampling field logbook and signed each page. Data obtained on all the samples was entered into the logbook, including sample identification and location, date, and time of collection, specified analytical method, field measurement and calibration data, field lot control number, and field observations. Any corrections in the logbooks were made by striking out the

incorrect entry using a single line. The person correcting the entry also initialed and dated the change. The correct entry was made immediately below the crossed-out entry.

Chain-of-Custody Forms. Chain-of-custody forms were used to record all samples collected and were shipped together with each sample shipment. The appropriate data for each sample was entered on the chain-of-custody form at the time of sampling.

Laboratory Logbook and Records. Following sample receipt at the laboratory, the sample custodian or laboratory personnel clearly documented the processing steps that were applied to the sample, in accordance with Contract Laboratory Program (CLP) laboratory requirements. The analytical data from laboratory QC samples were identified with each batch of related samples. The laboratory logbook also included the time, date, and name of the person who logged each sample into the laboratory system. This documentation was thorough enough to allow tracking of the sample analytical history without aid from the analyst. At a minimum, laboratory documentation procedures provided the following:

1. Recording in a clear, comprehensive manner using indelible ink.

2. Corrections to data and logbooks made by drawing a single line through the error and initialing and dating the correction.

3. Consistency before release of analytical results by assembling and cross-checking the information on the sample tags, custody records, bench sheets, personal and instrument logs, and other relevant data to verify that data pertaining to each sample were consistent throughout the record.

4. Observations and results (data) identified with the project number, date, and analyst and reviewer signatures on each line, page, or book as appropriate

5. Data recorded in bound books or sleaves of numbered pages, instrument tracings or hard copy, or microcomputer hard copy

6. Data tracking through document consolidation and project inventory of accountable documents, such as sample logbook, analysis data book, daily journal, instrument logbook, and narrative and numerical final reports.

5.0 Technology Performance Evaluation

This section provides an evaluation of the bioreactor performance. The evaluation includes a presentation of the analytical data collected, mass balance calculations performed, and an analysis of the overall system performance. The demonstration objectives were presented in Section 2.2, and are restated below:

- Determine the effectiveness of biotreatment processes for the treatment of SVE off-gases containing halogenated and nonhalogenated organic compounds.
- Quantify the total VOC mass removed by the process and compare observed DREs with the target DRE of 95%.
- Assess the treatment system performance.
- Obtain the data necessary to determine the cost to scale up the technology.

After the reactors and the associated equipment and accessories were assembled and installed and all of the utility hookups were completed, the system shakedown was performed. The initial startup and shakedown of the system began November 3, 1997. The final hook-up to the process gas stream from the SVE system was not made until the operation of the system was tested with ambient air.

The on-site GC was set up and calibrated with a combination gas purchased from Praxair, Inc. during system startup and shakedown. The combination contained the following halogenated and nonhalogenated contaminants of interest: PCE, TCE, 1,2-*cis*-DCE, 1,1,1-TCA, 1,1-DCA, and toluene. A second gas cylinder contained VC and was used to calibrate the GC for VC. These compounds were included in the gas standards because they persisted in the SVE process gas at measurable concentrations, according to the analysis of the site characterization data. For simplicity, the process performance was evaluated based on the removal of these compounds, as determined using the off-site analytical data.

5.1 Performance Data

Process gas samples were collected routinely from the biofilter influent gas stream (BFI), the biofilter effluent/CSTR influent gas stream (BFE/CSTRI), and the CSTR effluent gas stream

(CSTRE) for on-site analyses. The VOC concentrations were analyzed using the on-site GC. GC area counts were compared against known standards obtained from the calibrated gas standard for TCE, PCE, 1,1,1-TCA, 1,2-*cis*-DCE, 1,1-DCA, VC, and toluene. These data were used to obtain frequent and rapid analysis of the process gas stream for on-site operation of the biological treatment system. The data were not used to assess system performance.

During the field study, both CSTR liquid samples and the process gas samples from the BFI, BFE/CSTRI, and CSTRE were taken, and then were sent for off-site analyses, in accordance with the methods described in Section 4. Six off-site sample sets were taken on the following dates: December 3, 1997; December 23, 1997; January 13, 1998; January 30, 1998; March 2, 1998; and April 16, 1998. The December 3, 1998 samples were taken during the abiotic test period; all other samples were taken during normal operation of the biological treatment system. The vapor samples were collected in certified cleaned Summa canisters, and were sent to Air Toxics, Inc. (Folsom, California) for U.S. EPA TO-14 and TO-12 analyses; laboratory reports are provided in Appendix E. The liquid samples taken for VOC analysis were collected in 40-mL VOA vials and contained no headspace. Additional liquid samples were taken to measure TDS, TSS, COD, chloride (Cl^-) content, and total volatile solids (TVS). Sequoia Analytical Laboratories (Redwood, California) analyzed the liquid samples. The laboratory reports for the liquid analysis are presented in Appendix F.

The U.S. EPA TO-14 and TO-12 results for the contaminants of interest are shown in Tables 5-1 and 5-2, respectively. Aqueous VOC results are shown in Table 5-3, and the TDS, TSS, COD, Cl^-, and TVS results are shown in Table 5-4. The results shown in these tables are discussed in the following sections.

5.1.1 Abiotic Testing

The initial startup and shakedown were completed November 17, 1997. Afterwards, the final hookup to the process gas stream from the SVE system was made and the abiotic testing of the two-stage reactor process commenced. The connection was made from a blank pipe flange in the SVE off-gas manifold, provided by the Air Force. The vapor effluent from the biological treatment system was discharged to the atmosphere after treatment through the two in-series carbon canisters during the demonstration. The effluent from the first canister was monitored daily using a portable PID and the on-site GC to assess if breakthrough of VOC contaminants had occurred.

Table 5-1. Results for U.S. EPA Method TO-14 Analysis for the Reactor System at McClellan AFB

Sample Date	Sample ID	Contaminant Concentration (ppbv)						
		VC	1,1-DCA	1,2-*cis*-DCE	1,1,1-TCA	TCE	PCE	Toluene
12/03/97	BFI-V-144	880	1,600	2,400	6,600	6,400	3,500	4,100
	CSTRI-V-N142	950	1,700	2,500	6,900	6,700	3,700	4,200
	CSTRE-V-N141	950	1,700	2,600	7,000	6,800	3,800	4,300
12/23/97	BFI-V-154	900	2,700	2,800	30,000	18,000	20,000	7,600
	CSTRI-V-N156	140	420	440	4,700	2,800	3,000	1,100
	CSTRE-V-N157	1.8	5.4	2.2	68	43	59	5.9
01/13/98	BFI-V-144	230	3,000	1,000	32,000	17,000	21,000	8,800
	CSTRI-V-N142	150	1,800	610	19,000	9,800	12,000	4,400
	CSTRE-V-N141	130	1,600	240	18,000	8,400	11,000	1,300
01/30/98	BFI-V-N295	240	3,800	1,300	34,000	17,000	22,000	9,300
	CSTRI-V-N296	83	1,200	420	11,000	6,000	7,400	2,700
	CSTRE-V-N297	91	1,400	480	12,000	6,200	7,700	2,900
03/02/98	BFI-V-N349	310	3,200	1,100	28,000	14,000	21,000	7,600
	CSTRI-V-N348	160	1,400	500	13,000	6,200	8,800	2,800
	CSTRE-V-N347	130	1,400	180	13,000	5,800	9,500	920
04/06/98	BFI-V-N457	1,200	3,800	3,800	27,000	17,000	19,000	8,000
	CSTRI-V-N456	420	1,100	970	8,900	4,400	4,600	1,500
	CSTRE-V-N455	500	740	530	9,600	3,800	4,700	680

Table 5-2. Results for U.S. EPA Method TO-12 Analysis for the Reactor System at McClellan AFB

Sample Date	Sample ID	TNMOC TO-12 (ppmv) Reported as Heptane
12/23/97	BFI-V-N154	400
	CSTRI-V-N156	63
	CSTRE-V- N157	1.3
1/13/98	BFI-V-N215	340
	CSTRI-V-N214	190
	CSTRE-V- N213	130
1/30/98	BFI-V-N295	500
	CSTRI-V-N296	160
	CSTRE-V- N297	180
2/27/98	BFI-V-N349	850
	CSTRI-V-N348	150
	CSTRE-V- N347	140
4/3/98	BFI-V-N457	520
	CSTRI-V-N456	160
	CSTRE-V- N455	180

Table 5-3. CSTR Liquid Samples Results for the Compounds of Interest

Sample Date	Sample ID	Compounds Concentration (µg/L)						
		VC	1,1-DCA	1,2-*cis*-DCE	1,1,1-TCA	TCE	PCE	Toluene
12/03/97	CSTRE-L-N143	<0.50	23	49	39	67	25	52
12/29/97	CSTRE-L-N171	<0.50	<0.50	<0.50	<0.50	<0.50	0.8	<0.50
01/14/98	CSTRE-L-N222	<2.5	19	3.7	80	61	54	8.2
01/28/98	CSTRE-L-N283	<3.6	14	13	70	58	52	27
03/02/98	CSTRE-L-N353	<0.50	9.1	7.6	34	31	27	<0.50

ND = Not detected.

Table 5-4. CSTR Liquid Sample Results for Field Parameters

Sample Date	Sample ID	TDS (mg/L)	TSS (mg/L)	COD (mg/L)	Chloride (mg/L)	Total Volatile Solids (mg/L)
12/29/97	CSTRE-L-N168,N169,N170	3,100	3,100	2,800	100	810
01/06/98	CSTRE-L-N179,N180,N181	1,500	650	1,700	49	290
01/14/98	CSTRE-L-N224,N225,N226	1,800	850	770	53	490
01/19/98	CSTRE-L-N243,N244,N245	1,800	1,000	2,200	57	370
01/28/98	CSTRE-L-N280,N281,N282	1,700	2,900	1,000	60	3,300
03/02/98	CSTRE-L-N355,N356,N357	4,150	2,210	770	58	3,800
03/09/98	CSTRE-L-N387,N388,N389	3,200	2,700	2,600	NA	4,400
04/01/98	CSTRE-L-N441,N442,N443	4,500	4,300	1,600	160	6,500
04/08/98	CSTRE-L-N441,N442,N443	5,700	4,600	820	NA	6,000

NA = Not analyzed.

Abiotic testing was conducted at a process gas flowrate of approximately 9 scfm and continued until December 4, 1997. The temperature of the two-stage system was approximately 46.5°F in the biofilter and 63°F in the CSTR. The operating pressure of the two-stage reactor system was approximately 2.5 pounds per square inch gauge (psig). System losses were assessed using the on-site GC analyses of process gas samples taken from BFI, BFE/CSTRI, and CSTRE process gas streams. Once satisfactory performance was achieved in the field, the abiotic testing performance was verified by the off-site analysis of the December 3, 1997 vapor samples taken from the BFI, BFE/CSTRI, and CSTRE, and a liquid sample taken from the CSTR.

Reactor losses in the process gas stream during abiotic testing are shown in Table 5-5. Total losses for the seven target VOCs were !4.6% for the biofilter, !1.9% for the CSTR, and !6.6% overall. Negative losses do not indicate the production of VOCs in the reactors. Rather, they are most likely due to short-term concentration changes in the influent SVE off-gas stream.

A sudden VOC concentration drop in the reactor influent stream could result in negative losses if the drop results in lower influent concentrations than reactor effluent concentrations. Another possible reason for the negative losses is analytical error. However, errors in losses due to analytical error would be expected to vary between positive and negative values. The fact that all the losses are 0% or negative suggests that the reason for the negative losses is not analytical error, and is more likely due to VOC concentration changes in the influent process gas stream. These very low system losses, resulting from leaks or sorption onto reactor walls, were insignificant.

Table 5-5. Abiotic Losses

	Percent Losses (%)		
Compound	**Biofilter**	**CSTR**	**Total**
PCE	!2.7	!5.7	!8.6
TCE	!1.5	!4.7	!6.3
1,2-*cis*-DCE	!4.0	!4.2	!8.3
1,1,1-TCA	!1.4	!4.5	!6.1
1,1-DCA	0.0	!6.3	!6.3
VC	0.0	!8.0	!8.0
Toluene	!2.4	!2.4	!4.9

5.1.2 Reactor Startup

Upon the completion of the abiotic testing period, the CSTR was inoculated with the G-4 bacteria (shipped on ice overnight to the site by Envirogen and thawed before being added to the CSTR), and the growth period of the bacteria was started. Phenol, ammonium nitrate (NH_4NO_3), and monopotassium phosphate (KH_2PO_4) were added to the reactor to facilitate growth of the G-4 bacteria enzymes, which were used in the CSTR to degrade the non-PCE chloroethene off-gas contaminants. The 1.5-L solutions of NH_4NO_3 and KH_2PO_4 added to the reactor resulted in initial concentrations of 500 and 60 mg/L, respectively. Approximately 35 L of G-4 bacteria was added to the reactor.

The CSTR was operated in batch mode for approximately 2 weeks, with regular batch phenol and nutrient feeding. The batch mode allowed for the growth of the G-4 bacteria without washout in the liquid wastestream and without potential toxicity from the process gas stream.

Phenol was used as the food source for the G-4 bacteria, and phenol degradation rates were measured to determine phenol addition requirements. Three phenol degradation tests were performed during the first week of batch mode operation. To determine phenol concentration in the CSTR, phenol concentrations in the CSTR aqueous phase were measured using a phenol assay kit (Hach®; Loveland, Colorado). The average phenol degradation rate was 0.47-mg phenol/L/min, and the phenol addition rate was established at 846-mL per hour. The addition rates of the NH_4NO_3 and KH_2PO_4 were set at 3 gallons and 1 gallon per hour (gph), respectively. Phenol and nutrient addition rates were closely monitored during the batch mode of operation, so that the G-4 bacteria would not be under or oversupplied with phenol, or nutrients. The process parameters, such as phenol and nutrient concentrations, and optical density (OD), pH, and protein concentrations, were measured weekly when the reactor system was in operation. The results of these analyses are presented in tabular form in Appendix G.

The batch mode of operation continued until December 17th, 1997, at which time the biofilter was loaded with a general inoculum (Phenobac®; Polybac Corporation, Bethlehem, Pennsylvania) and continuous operations were started. Contaminated air from the SVE system was introduced to the biofilter unit and flowed upward through the medium. The medium compartment was constructed with two sets of irrigation hoses to maintain the 60 to 70% medium moisture levels, which were required for optimum operating conditions. In addition to providing moisture, the upper irrigation hoses were used to provide inorganic soluble nutrients (nitrogen and phosphorus) at the beginning of the experiment. 1.5-L solutions of NH_4NO_3 and KH_2PO_4 were added to the reactor, resulting in initial concentrations of 500 and 60 mg/L, respectively. The lower humidification zone ensured that the air entering the biologically active area of the biofilter was saturated with water vapor. A sample of the biomass was taken for analysis at Microbial Insights, Inc. (Knoxville, Tennessee). The biomass underwent PLFA analysis, which included metabolic status ratio and community structure percentage. The results of the biomass analysis performed are presented in Table 5-6. The laboratory reports on the biomass analysis are included as Appendix G.

The vapor effluent from the biofilter was then piped to the CSTR for the second stage of contaminant removal. The startup and acclimation phase of the demonstration was performed in order to reach steady-state operation. The liquid level in the tank was maintained at approximately 72 in. by adding fresh potable water at 1 gph to the CSTR. The water in the tank passed

over a weir in the tank and was discharged to the waste holding tank. The operating pH of the CSTR was maintained at a pH of 7.0 by pumping a 25% NaOH solution into the reactor. During the demonstration startup and acclimation phase, samples were taken to perform a mass balance on the contaminants of interest to determine DREs. Vapor-phase samples were collected from the BFI, BFE/CSTRI, and CSTRE streams, and liquid-phase samples were collected from the CSTR effluent stream for the mass balance calculations (see Tables 5-1 through 5-4); vapor samples were sent to Air Toxics, Ltd. (Folsom, California) and liquid samples were sent to Sequoia Analytical (Redwood City, California) for analyses. On-site vapor samples were taken using Tedlar™ bags for analysis by the on-site GC. The on-site GC results are shown in Appendix D.

Table 5-6. Biofilter Biomass Analytical Results

Parameter	Sample ID	
	BFI-S-N148	BFI-S-N498
Sample Date	12/17/97	4/15/98
Weight of wet soil	39.50 g	40.03 g
Moisture content	62%	66%
Weight of dry soil	14.84 g	13.77 g
Total Picomoles of PLFA	1,679,898	2,454,657
Picomoles total PLFA/g dry soil	113,169	178,293
Cells/g dry soil	2.26 E+09	3.57 E+09
Picomoles prokaryote PLFA	85,771	150,248
Picomoles eukaryote PLFA	27,432	28,028
Ratio prokaryote/eukaryote	3	5
Metabolic Status: (Ratio)		
cy17:0/16:1w7c	0.27	0.69
cy19:0/18:1w7c	0.54	0.54
Total cy17/cy19	0.81	1.23
16:1w7t/16:1w7c	0.02	0.13
18:1w7t/18:1w7c	NC	0.03
Total cy16/cy18	0.02	0.16
Community Structure: (% of Total PLFA)		
TerBrSats	13.4	16.7
Monos	38.8	39.2
BrMonos	5.5	5.3
MidBrSats	5.2	7.0
NSats	12.9	16.1
Eukaryotes	24.2	15.7

NC = Not calculated.

Overall DREs on the December 23, 1997 sampling date were approximately 80% for the biofilter, 95% for the CSTR, and more than 99% overall. These high DREs are attributed to contaminant sorption onto the biofilter medium and CSTR biomass during startup. The target removal percentage of 95% was achieved only during the December 23, 1997 sampling event, and much lower DREs were observed thereafter.

5.1.3 Performance Monitoring

The performance monitoring phase of the project began January 13, 1998, and continued through April 15, 1998. The maximization of vapor throughput and contaminant DREs was attempted by adjusting vapor flowrates or phenol addition rates, with no success at achieving the 95% DRE goals.

During the 4-month operating period, the SVE system experienced repeated mechanical shutdowns, reducing the net operating time to 60 to 65%. Most of the downtime experienced during this demonstration was caused by the SVE system, which fed the contaminated vapor stream to the biofilter. The downtime experienced during this project that was directly attributable to the operation of the bioremediation system was mostly due to inoculation/reinoculation of the system. Each inoculation period lasted approximately 2 weeks. At previous locations where this type of technology was demonstrated, normal downtimes ranged from 3% (Robins AFB) to 11% (F.E. Warren AFB). Sample dates for off-site analyses targeted periods when the SVE system was operational.

The CSTR was reinoculated on February 10 and again on March 12, 1998. Preliminary field results showed unsatisfactory TCE degradation rates in the CSTR, so the CSTR was reinoculated on the aforementioned dates to stimulate G-4 growth and TCE degradation. Prior to reinoculation, liquid samples of the CSTR effluent, on-site chemicals, and on-site potable water had been sent to Envirogen's laboratories for analysis of G-4 bacteria and TCE degradation. At Envirogen it was determined that TCE was not effectively degraded by the phenol-degrading bacteria in the CSTR, and that G-4 constituted only a small fraction of these bacteria, further justifying the reinoculation events. The reinoculation procedure for the CSTR was performed as described below:

1. Drained the contents of the CSTR.
2. Cleaned the inside of the reactor with fresh water (3 times).

3. Refilled the CSTR with fresh (potable) water, and added the following ingredients to approximately 1,000 gal of water:
   - 5,678 g KH_2PO_4 (1.5 g/L)
   - 4,242 g NH_4NO_3 (1.1 g/L)
   - 567 g magnesium sulfate heptahydrate (0.15 g/L)
   - 3.55 L 800x Trace Metal Solution
4. Elevated the pH to approximately 7.0 with caustic solution
5. Added 380 mL phenol to the reactor
6. Added 18.7 L G-4 bacteria to the reactor.

During the March 12, 1998 reinoculation event, a water purification system was installed so the potable water source could remove any potentially inhibitory contaminants. The procedure for the second reinoculation was very similar to that of the first, except that the potable water source was treated before it entered the CSTR, using the GAC/ion exchange treatment system that was installed.

Following each reinoculation, the system was operated in batch mode for 10 days. During this time the biofilter process gas was diverted from the CSTR, and only ambient air was directed to the CSTR. The ambient air flowrate through the reactor was 8.0 scfm. During batch mode the phenol degradation rates were monitored, as with the initial inoculation. Phenol was metered at 470 mg/L/min, similar to the initial inoculation.

Figures 5-1 through 5-7 show the calculated gas-phase removal efficiencies for the biofilter reactor and CSTR, and also show the total removal efficiency for PCE, TCE, 1,2-*cis*-DCE, VC, 1,1,1-TCA, 1,1-DCA, and toluene. Removal efficiencies also are shown in Table 5-7. During abiotic testing, system losses were less than 10% (see Section 5.1.1).

Total DREs were close to 100% during startup. The fact that DREs dropped over time indicates that these DREs most likely represent contaminant partitioning onto the biofilter medium and the CSTR biomass, and are not due solely to contaminant biodegradation. Negative DREs do not suggest VOC production in the reactors and are likely due to short-term fluctuations in the SVE off-gas, (i.e., influent process gas) VOC concentrations, where influent concentrations were temporarily lower than effluent concentrations.

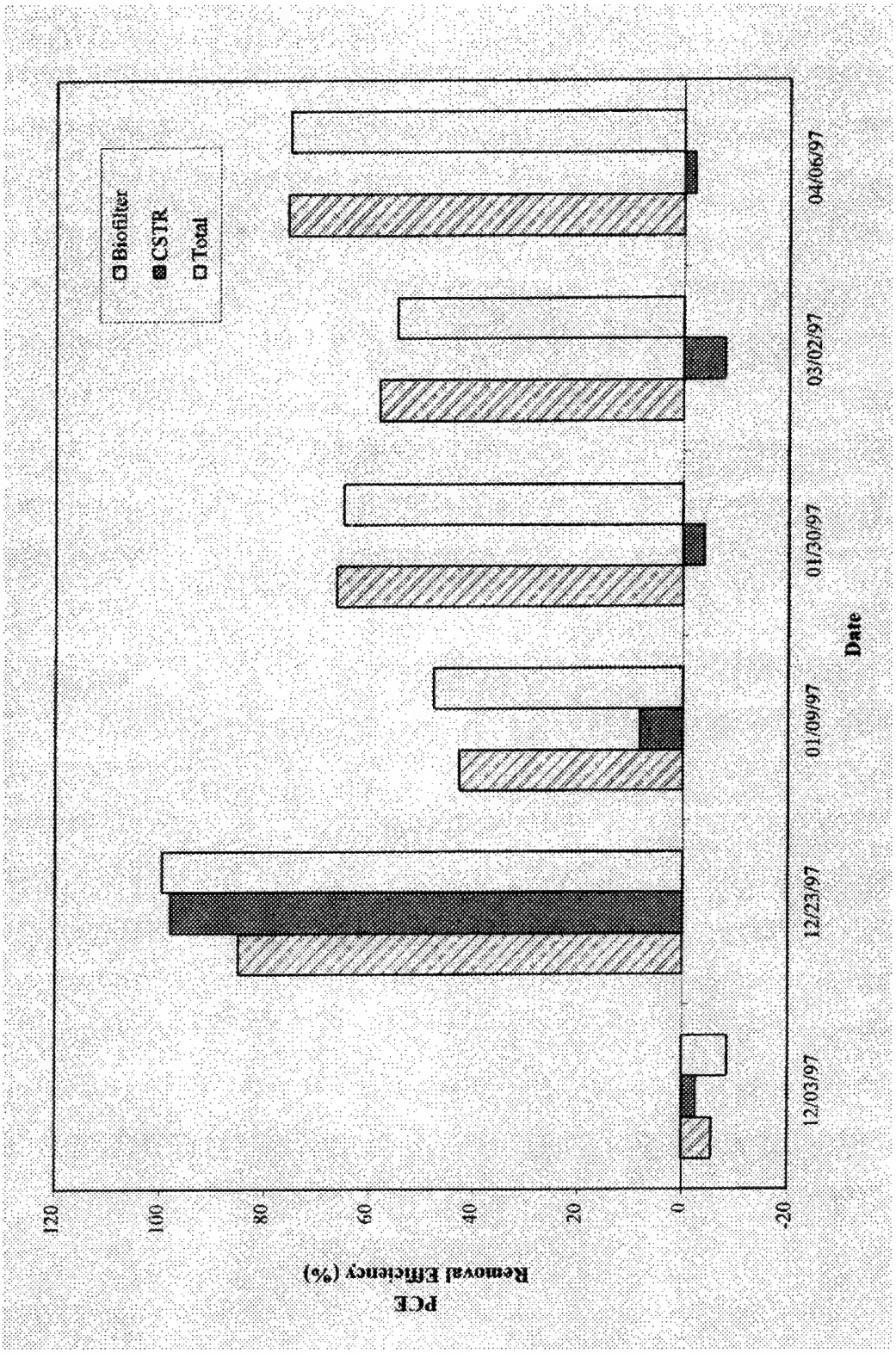

Figure 5-1. Calculated PCE Gas-Phase Removal Efficiencies

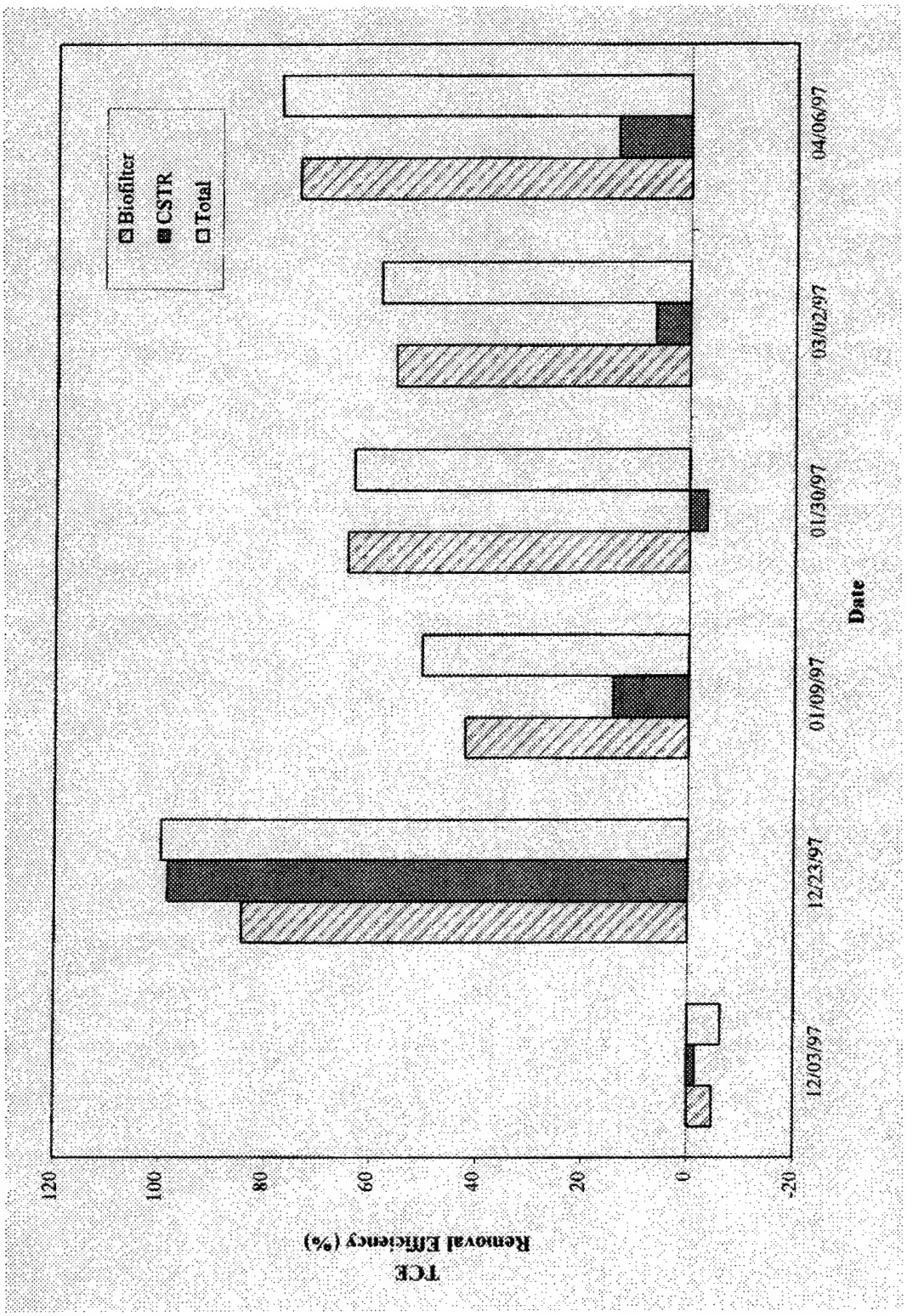

Figure 5-2. Calculated TCE Gas-Phase Removal Efficiencies

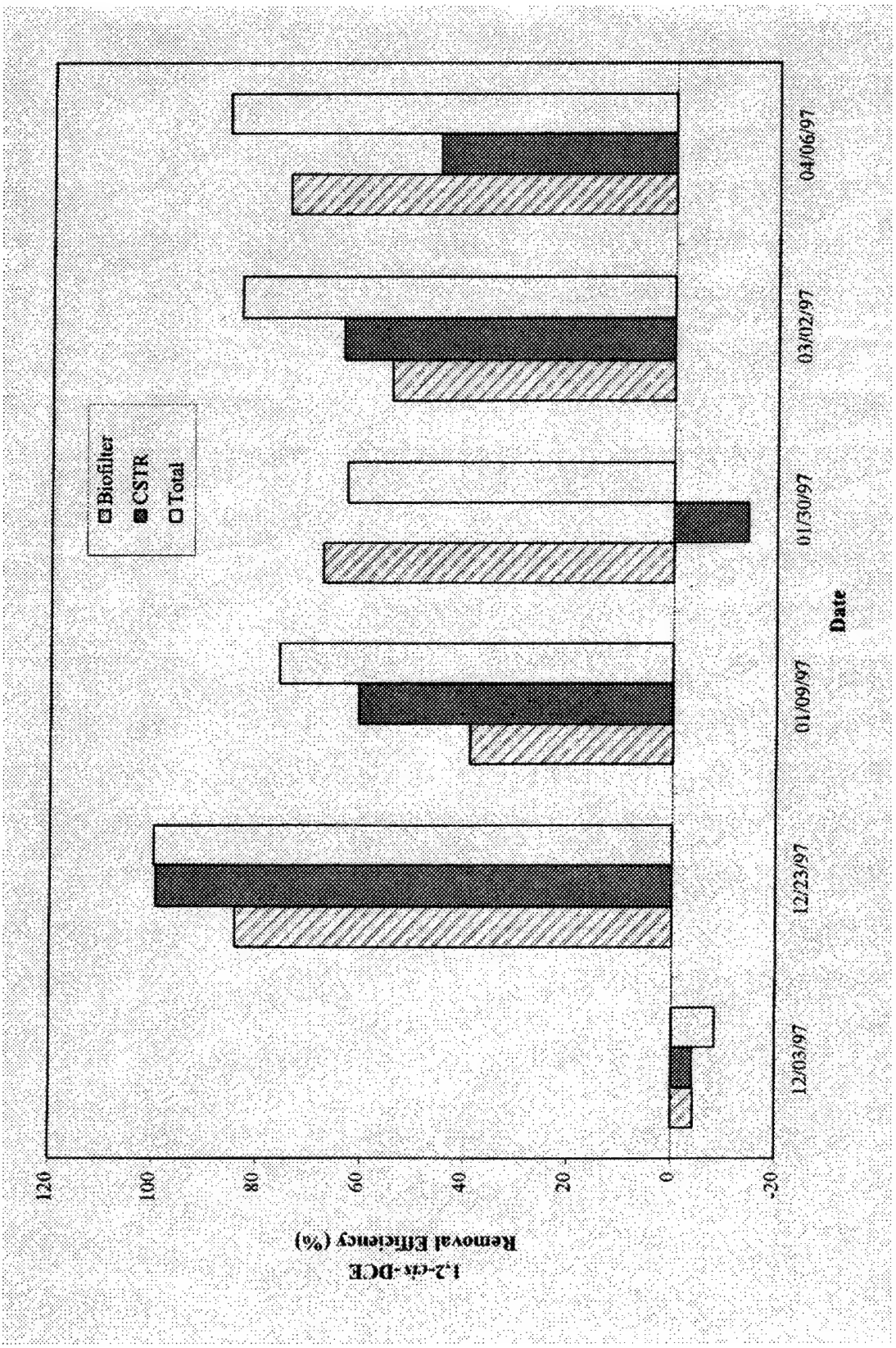

Figure 5-3. Calculated 1,2-*cis*-DCE Gas-Phase Removal Efficiencies

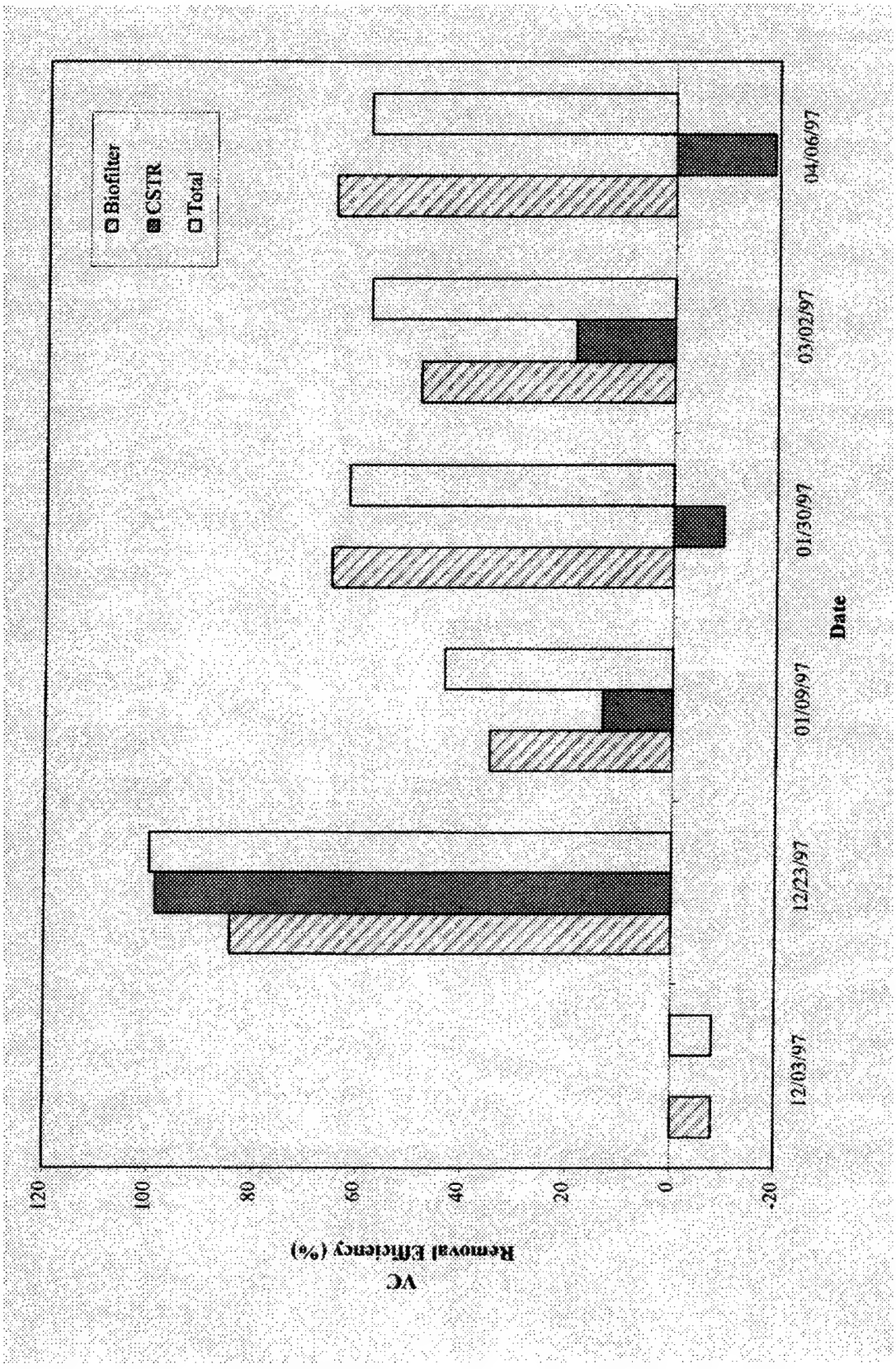

Figure 5-4. Calculated VC Gas-Phase Removal Efficiencies

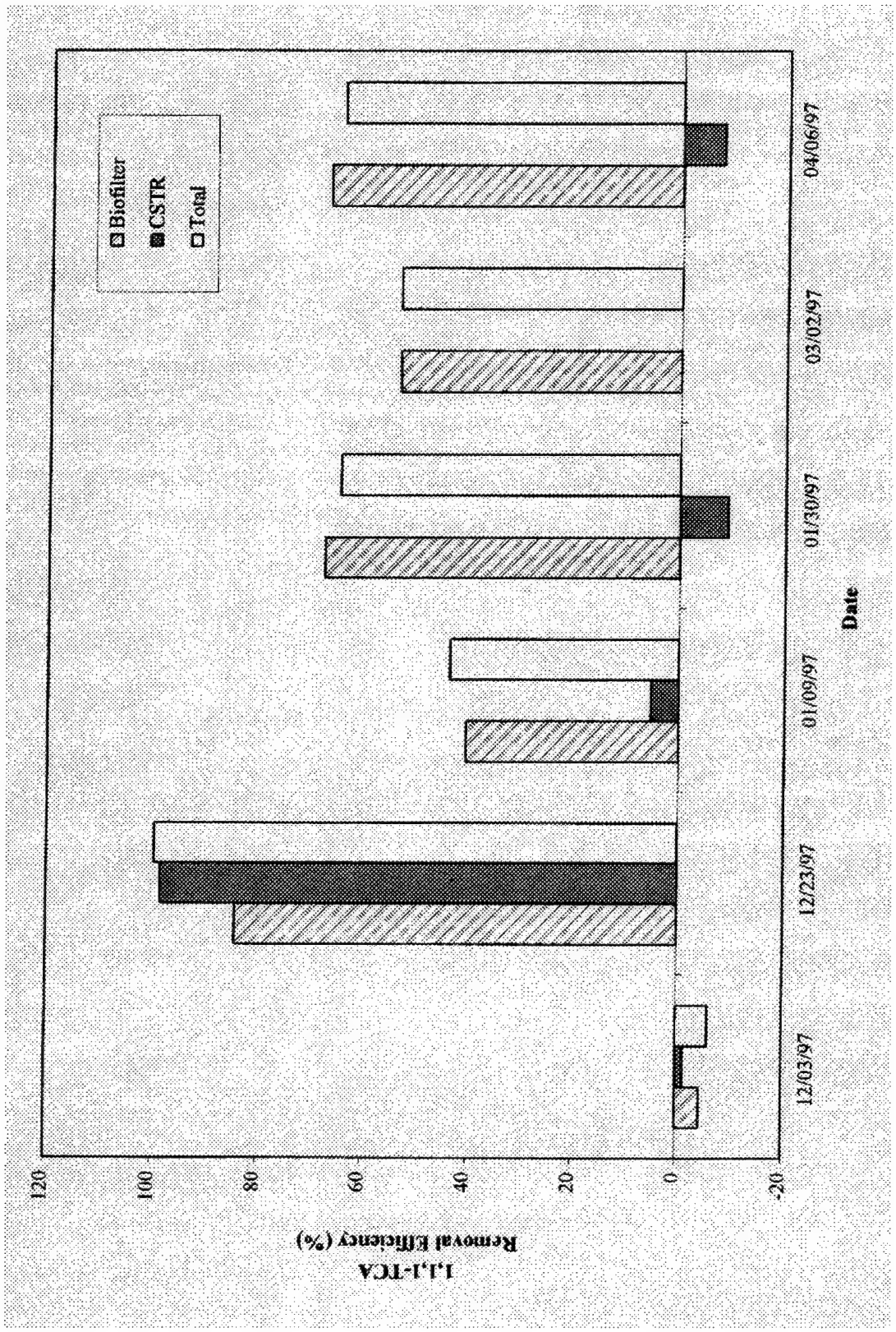

Figure 5-5. Calculated 1,1,1-TCA Gas-Phase Removal Efficiencies

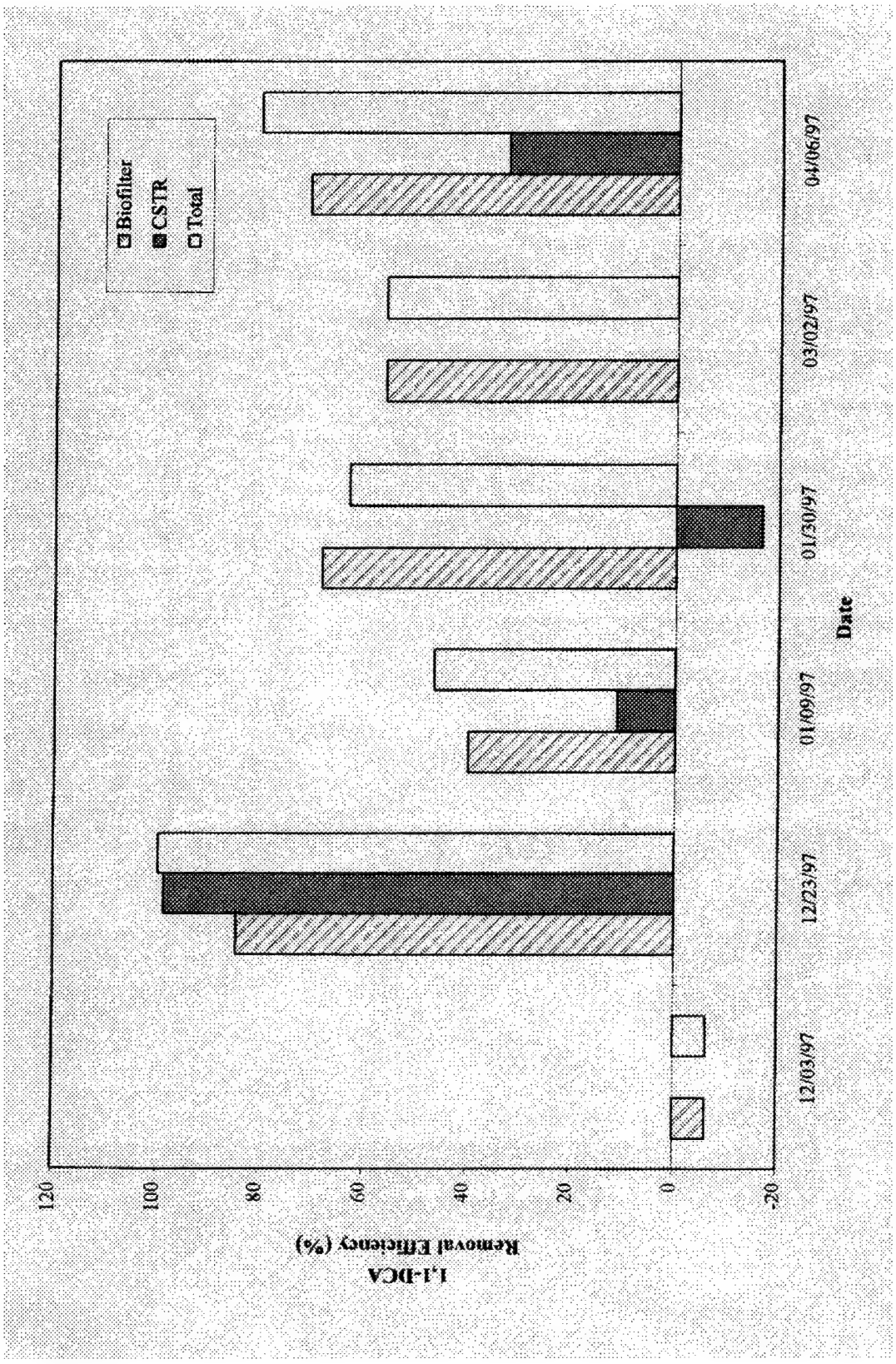

Figure 5-6. Calculated 1,1-DCA Gas-Phase Removal Efficiencies

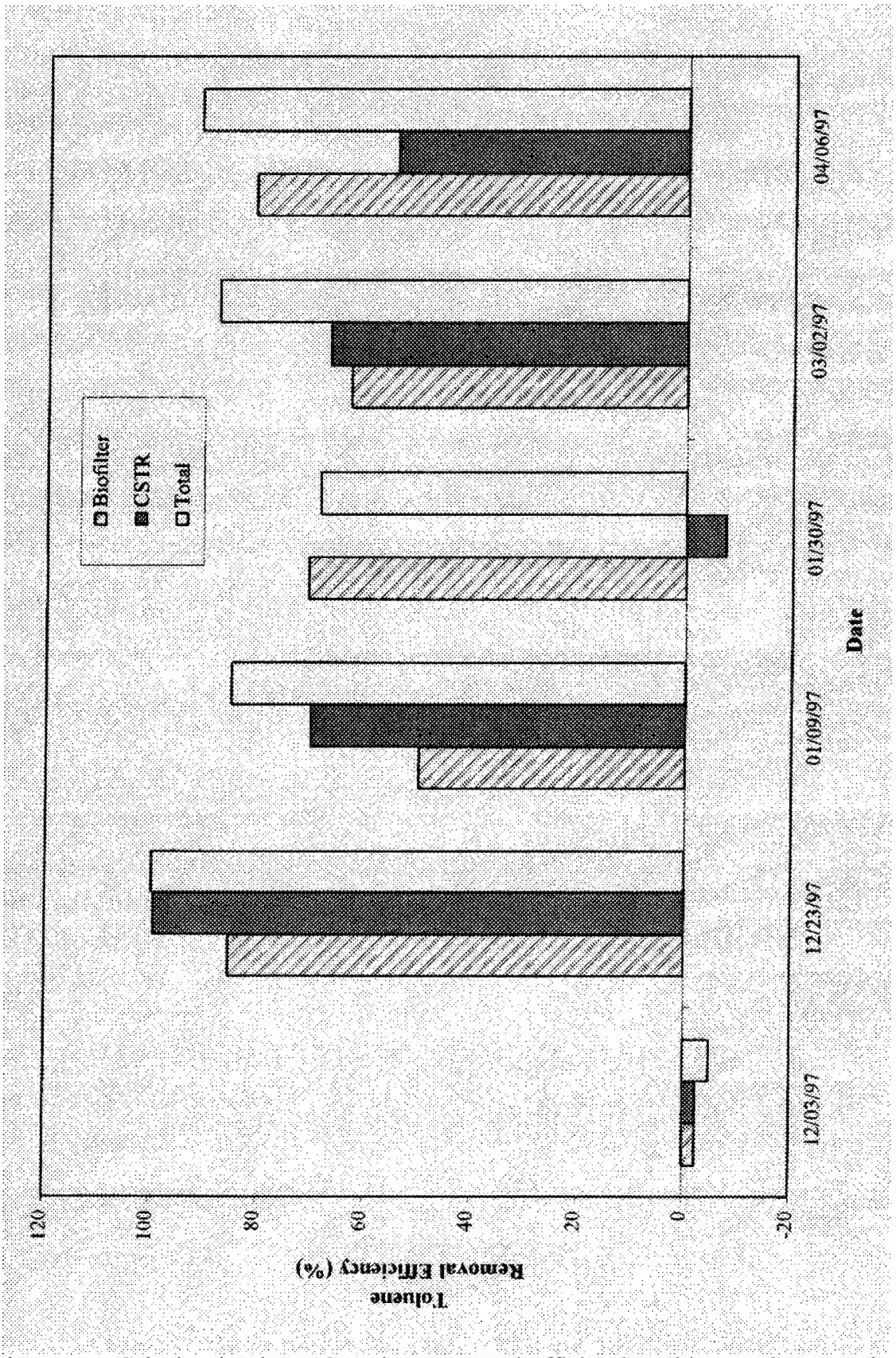

Figure 5-7. Calculated Toluene Gas-Phase Removal EfficienciesTable 5-7. Compound

Table 5-7. Compound Gas-Phase Removal Efficiencies

Compound	Off-Site Sample Date	Gas-Phase Removal Efficiency (%)		
		Biofilter	CSTR	Total
PCE	12/03/97 (abiotic)	-5.7	-2.7	-8.6
	12/23/97 (startup)	85	98	100
	01/13/98	43	8.3	48
	01/30/98	66	-4.1	65
	03/02/98	58	-8.0	55
	04/06/98	76	-2.2	75
TCE	12/03/97 (abiotic)	-4.7	-1.5	-6.3
	12/23/97 (startup)	84	98	100
	01/13/98	42	14	51
	01/30/98	65	-3.3	64
	03/02/98	56	6.5	59
	04/06/98	74	14	78
1,2-*cis*-DCE	12/03/97 (abiotic)	-4.2	-4.0	-8.3
	12/23/97 (startup)	84	100	100
	01/13/98	39	61	76
	01/30/98	68	-14	63
	03/02/98	55	64	84
	04/06/98	74	45	86
VC	12/03/97 (abiotic)	-8.0	0.0	-8.0
	12/23/97 (startup)	84	99	100
	01/13/98	35	13	43
	01/30/98	65	-10	62
	03/02/98	48	19	58
	04/06/98	65	-19	58
1,1,1-TCA	12/03/97 (abiotic)	-4.5	-1.4	-6.1
	12/23/97 (startup)	84	99	100
	01/13/98	41	5.3	44
	01/30/98	68	-9.1	65
	03/02/98	54	0.0	54
	04/06/98	67	-7.9	64
1,1-DCA	12/03/97 (abiotic)	-6.3	0.0	-6.3
	12/23/97 (startup)	84	99	100
	01/13/98	40	11	47
	01/30/98	68	-17	63
	03/02/98	56	0.0	56
	04/06/98	71	33	81
Toluene	12/03/97 (abiotic)	-2.4	-2.4	-4.9
	12/23/97 (startup)	86	99	100
	01/13/98	50	70	85
	01/30/98	71	-7.4	69
	03/02/98	63	67	88
	04/06/98	81	55	92
Total (Based on the total mass of the contaminants of interest)	12/03/97 (abiotic)	-4.6	-1.9	-6.6
	12/23/97 (startup)	85	99	100
	01/13/98	43	15	51
	01/30/98	67	-6.8	65
	03/02/98	56	5.9	59
	04/06/98	73	6.1	74

5.1.3.1 PCE DREs

Between January13 and April 6, 1998, the PCE DREs ranged from 43 to 76% in the biofilter, and were close to zero in the CSTR; total DREs ranged from 48 to 75%. The negligible DREs in the CSTR are consistent with the fact that PCE is not degraded aerobically under cometabolic conditions or as a direct substrate.

The higher DREs in the biofilter were unexpected, once again, because PCE is not aerobically degraded. These DREs could have been as a result of PCE partitioning onto the biofilter medium, in part. However, it is unlikely that PCE partitioning alone would account for 40 to 70% removals over the 4-month operating period. Furthermore, under partitioning alone, PCE DREs would be expected to decrease as sorption sites become filled and PCE breakthrough occurs. In the biofilter, on the other hand, removal efficiencies appeared to increase over time, which is characteristic of increased biomass and biodegradation. PCE biodegradation could have been possible if anaerobic conditions existed in the biofilter. Such conditions are possible under high organic loading conditions that create micro-anaerobic zones where oxygen may be mass-transfer limited, resulting in anaerobic conditions where dechlorination could occur. The results of this demonstration are insufficient to establish the PCE removal mechanism.

5.1.3.2 TCE DREs

The TCE DREs in the biofilter were very similar to PCE DREs; the same removal mechanisms may have occurred for both VOCs. Unlike PCE, TCE can be degraded aerobically under cometabolic conditions. In the biofilter, toluene could have acted as the cometabolic cosubstrate; toluene is a known cosubstrate for TCE cometabolism.

In the CSTR, TCE degradation was slightly higher than PCE degradation during the January 13 and April 6 sampling events, as indicated by the slightly higher DREs. However, the TCE DREs in the CSTR were much lower than expected, and were insufficient to meet the 95% overall target DRE. The low TCE DREs in the CSTR attest to the failure of the cometabolic process for TCE degradation. The reason for this failure was investigated, but remains unknown.

5.1.3.3 1,2-*cis*-DCE DREs

The DCE DREs ranged from 39 to 74% in the biofilter. Except for the January 30 CSTR DREs, which were negative, DREs in the CSTR ranged from 45 to 64%. Total DREs ranged from 63 to 86%. The 1,2-*cis*-DCE DREs in the biofilter were comparable to those for PCE and TCE. However, for the CSTR, they were much higher than PCE and TCE. These much higher DREs were not unexpected, because DCE can be degraded aerobically as a direct substrate for carbon and energy or cometabolically using phenol as a cosubstrate. The field results did not differentiate the DCE degradation mechanism. Total DCE DREs approached but did not achieve the 95% target DREs.

5.1.3.4 VC DREs

The VC DREs ranged from 35 to 65% in the biofilter and from less than zero to 19% in the CSTR. Total DREs ranged from 43 to 62%. The VC DREs in the biofilter were comparable to those for the other chloroethenes, and in the CSTR they were similar to the PCE and TCE DREs. Like 1,2-*cis*-DCE, VC can be degraded aerobically as a direct substrate for carbon and energy or cometabolically using phenol as a cosubstrate. The low VC DREs in the CSTR suggest that the effects of both mechanisms were minimal.

5.1.3.5 1,1,1-TCA DREs

The 1,1,1-TCA DREs in the biofilter ranged from 41 to 68% and were negligible in the CSTR. Total DREs ranged from 44 to 65%. Like TCE, 1,1,1-TCA degrades aerobically only under cometabolic conditions. However, it does not degrade with phenol as a cometabolic cosubstrate and requires alternative cosubstrates that were not supplied, such as methane, propane, or butane. Thus, 1,1,1-TCA degradation was not expected in the CSTR, and, not suprisingly, the 1,1,1-TCA DREs were comparable to those for TCE.

5.1.3.6 1,1-DCA DREs

The 1,1-DCA DREs in the biofilter ranged from 40 to 71% and from less than zero to 33% in the CSTR. Total DREs ranged from 47 to 81%. Like DCE and VC, 1,1-DCA may be degraded aerobically as a direct substrate or cometabolically. However, like 1,1,1-TCA, phenol

is not a suitable cosubstrate for 1,1-DCA degradation. The 33% DREs during the April 6 sampling event suggest that 1,1-DCA degraders were present toward the end of the study.

5.1.3.7 Toluene DREs

The toluene DREs ranged from 63 to 81% in the biofilter and, except for the January 30 data, from 55 to 70% in the CSTR. Toluene had the highest total DREs, which ranged from 69 to 92%. The lowest DRE was during the January 30 sampling event, when the DREs for all the contaminants of interest were lowest. These low DREs on January 30 could indicate an upset in the CSTR performance or could be due to unknown sampling or analytical errors. The toluene DREs approached but did not achieve the 95% target DRE.

Toluene was most likely the most degradable substrate examined, because it is easily degraded aerobically as a direct substrate for carbon and energy, does not depend on cometabolic conditions, and can be degraded in biofilters and in suspended-growth reactors.

5.1.3.8 Total DREs

Total DREs were calculated based on the sum of the contaminant mass from the target compounds listed in Table 5-7. Total DREs in the biofilter ranged from 43 to 73%, and from zero to 15% in the CSTR. The zero percent DREs in the CSTR were observed on January 30, 1998. Total DREs ranged from 51 to 74%, and did not meet the target DRE of 95%.

5.1.4 Mass Balances

Mass balances in the reactor system were calculated using Equation 5-1. For each process gas sampling period, liquid samples were taken from the CSTR. Off-site analytical data were used to calculate mass balances for the contaminants of interest. Tables 5-8, 5-9, and 5-10 show the mass balances for each sampling event for the two-stage reactor system, the biofilter unit, and the CSTR unit, respectively.

$$MB = [C_{post}/C_{pre} - (0.093)\,(F_l C_l)/(F_v C_{pre})]\,[100\%] \qquad \text{(Equation 5-1)}$$

where: MB = system mass balance

C_{post} = effluent vapor concentration (mg/m^3)

C_{pre} = influent vapor concentration (mg/m^3)

C_l = effluent liquid concentration (mg/L)

F_l = effluent liquid discharge rate (gpd)

F_v = vapor flowrate (cfm)

0.093 = conversion factor for: $\frac{gpd * mg/L}{cfm * mg/m^3}$.

For the purposes of the mass balance calculations, the water effluent flowrate from the CSTR to the water discharge storage tank was assumed to be 1.0 gph for each sampling event. The vapor flowrate was based on the average value for each individual sampling period.

The mass balances for each sampling event were performed to calculate the total amount of each contaminant of interest that was removed by the two-stage process. For all of the mass balance calculations, the masses of contaminants discharged in the liquid-phase effluent were negligible compared to the masses in the gas-phase effluent. For this reason, the mass balances were very similar to the DREs, which were calculated based on the mass removed in the gas phase. As with the DREs calculated for the biofilter, CSTR, and total reactor process, mass balances were close to 100% on December 3, 1997 during the abiotic test, indicating that system losses were negligible. Mass balances were close to zero on December 23, 1997, during the startup phase, and were most likely a result of contaminant sorption onto the biofilter medium and the CSTR biomass. During normal system operation, between January 13 and March 2, 1998, the highest removals occurred in the biofilter. The CSTR showed a nearly 100% mass balance on these dates, indicating minimal mass destruction. Over the performance-monitoring period, the total system losses were less than 60% for the two-stage reactor process.

Table 5-8. Mass Balance Calculations for the Compounds of Interest for the Biofilter Unit

Sample Date	C_{post} (mg contaminant/ m^3)	C_{pre} (mg contaminant/ m^3)	C_l[a] (mg contaminant/ m^3)	F_l (gpd)	F_v (cfm)	Mass Balance (%)
12/03/97	26,650	25,480	0.0	24.0	8.0	105
12/23/97	12,600	64,000	0.0	24.0	8.0	19.7
01/13/98	47,760	83,030	0.0	24.0	8.0	58
01/30/98	28,803	87,640	0.0	24.0	8.0	33
03/02/98	32,860	75,210	0.0	24.0	6.5	44

(a) Reported concentration was measured to be below the detection limit.

Table 5-9. Mass Balance Calculations for the Compounds of Interest for the Continuously Stirred Tank Reactor

Sample Date	C_{post} (mg contaminant/ m^3)	C_{pre} (mg contaminant/ m^3)	C_i (mg contaminant/ m^3)	F_l (gpd)	F_v (cfm)	Mass Balance (%)
12/03/97	27,150	26,650	2.6 E-01	24.0	8.0	102
12/23/97	599.9	12,600	8.6 E-04	24.0	8.0	4.8
01/13/98	40,870	47,760	2.3 E-01	24.0	8.0	86
01/30/98	30,571	28,803	2.3 E-01	24.0	8.0	106
03/02/98	30,930	32,860	1.1 E-01	24.0	6.5	94

Table 5-10. Mass Balance Calculations for the Compounds of Interest for the Two-Stage Reactor System

Sample Date	C_{post} (mg contaminant/ m^3)	C_{pre} (mg contaminant/ m^3)	C_i (mg contaminant/ m^3)	F_l (gpd)	F_v (cfm)	Mass Balance (%)
12/03/97	27,150	25,480	2.6 E-01	24.0	8.0	107
12/23/97	599.9	64,000	8.6 E-04	24.0	8.0	0.3
01/13/98	40,870	83,030	2.3 E-01	24.0	8.0	49
01/30/98	30,571	87,640	2.3 E-01	24.0	8.0	35
03/02/98	30,930	75,210	1.1 E-01	24.0	6.5	41

5.1.5 Laboratory Investigations of Cometabolic TCE Degradation in the CSTR

On February 17, 1998, CSTR process water, phenol, and the trace metal solution (i.e., nutrient solution) were shipped to Envirogen laboratories for batch testing, to investigate TCE degradation by the CSTR biological culture. These laboratory tests were conducted to examine the health of the G-4 culture, and to establish whether the CSTR bacteria could degrade TCE.

Table 5-11 shows the experimental matrix and results of the first laboratory tests performed at Envirogen's laboratory. The experiments were carried out in 150-mL flasks, using 50 mL of site water or lab water for each flask. In some instances the site water was autoclaved prior to being added to the flasks. The industrial grade reagents used for the Basal Salt medium (BSM) solution were the same as the on-site nutrients. However, for the BSM B 800X solution, 1.25-mL of the BSM standard was added without the nitrilotriacetic acid trisodium salt and magnesium sulfate heptahydrate compunds. This was done to assess whether the poor field performance could be attributed to the types of nutrients used in the field. Also, either 45-μL of the 10% site phenol or 5-μL of a 20% laboratory phenol solution were added to each flask, to examine whether the

Table 5-11. Results of the Laboratory Batch Tests Performed at Envirogen

Test	Basic Salts	BSM B 800X	Water Used	Phenol Used	OD after 1 day	OD after 2 days	Phenol detected after 1 day	Phenol detected after 2 days	TCE Degradation Rate (μg/min/mg protein)
1	site	lab	site	lab	0.015	0.0	+	+	No degradation
2	site	lab	DI	lab	0.23	0.46	-	-	0.053
3	BSM[a]	BSM[a]	site	lab	0.011	0.0	+	+	No degradation
4	site	lab	site	lab	0.05	0.04	+	+	No degradation
5	site	lab	site	site	0.035	0.015	+	+	No degradation
6	2x site	2x lab	site	lab	0.016	0.21	+	-	0.084
7	BSM[a]	BSM[a]	DI	site	0.16	0.29	-	-	0.03
8	lab	lab	site	lab	0.025	0.019	+	+	No degradation
9	site	lab	site[b]	lab	0.25	0.55	-	-	0.041
10	site[c]	lab	site	lab	0.011	0.005	+	+	No degradation
11	site	2x lab	site	lab	0.04	0.175	+	-	0.058
12	site	2x lab	site	lab	0.041	0.021	+	+	No degradation
13	BSM[a]	BSM[a]	site[b]	lab	0.27	0.67	-	-	0.055

(a) BSM standard solution used, 2 g/L KH_2PO_4, 1.5 g/L NH_4NO_3, 0.2 g/L $MgSO_4 \cdot 7H_2O$.
(b) Site water was sterilized by autoclaving.
(c) Site basic salts were used, with the addition of 2 times the KH_2PO_4.

phenol grade could affect G-4 performance in the field. The pH of the flasks was adjusted to 7.0 to 7.1 with 2 M sodium hydroxide, and the bottles were inoculated with a laboratory-grown G-4 culture. After the culture was grown on the phenol, the suspension was centrifuged, washed twice with deionized water, and re-suspended in deionized water at an OD of 1.25. All flasks were closed with a foam stopper, and were then placed on a shaker table at 30°C.

The best phenol removals and highest TCE degradation rates were observed in bottles 2, 6, 7, 9, 11, and 13. These bottles used either deionized (DI) water, autoclaved site water, or untreated site water with 2 times BSM salts.

Table 5-12 summarizes specific TCE degradation rates and concentration tolerances of cosubstrate-degrading enrichments for comparison. In general, the rates measured during this demonstration were lower than the rates reported in Table 5-12. However, it is difficult to make a specific comparison of all the cultures because of their different growth and environmental conditions. In addition, pure cultures are expected to have much higher specific growth rates than mixed cultures because a much higher percentage of the normalized biomass concentration contributes directly to TCE degradation in pure cultures.

Table 5-12. Reported Maximum TCE Degradation Rates and TCE Concentrations Tested for Phenol

Culture	Reported TCE Degradation Rate (g/g VSS-d)	Highest TCE Concentration Degraded/Tested (mg/L)	Test Temperature (°C)	Reference
P. cepacia G4 PR1	0.09	>70, <428	23	Shields and Reagin (1992)
P. cepacia G4	1.1 (0.7)	50	28	Folsom and Chapman (1991)
P. cepacia G4	1.92	30	26	Folsom et al. (1990)

One possible conclusion from these results was that the potable water being used on site negatively affected the G-4 bacterial strain. The use of autoclaved water (Test #9) also showed some TCE degradation; however, in general, when site water was used in the laboratory no growth of the G-4 bacteria was observed. Other conclusions that were drawn from the laboratory tests were that the use of laboratory chemicals instead of the on-site chemicals had no apparent effect on bacteria growth. The growth of the bacteria with the laboratory chemicals and nonsterile site water showed the best results when double the concentration of nutrient solution was used.

A second set of batch tests was performed at Envirogen laboratories to determine the effects of the potable water on G-4 bacteria growth and contaminant degradation rates. Bottles were prepared in duplicate. Table 5-13 details the second set of batch test results. These studies suggested that water softening of the on-site potable water would have a positive effect on degradation rates and bacteria growth. The batch test results led to the use of a cation exchange unit (CEU) as part of the field demonstration. Potable water was first passed through a GAC filter and then through the CEU before it was pumped to the CSTR. However, this process modification failed to stimulate the growth for G-4 in the field, and the target DREs were not achieved.

Because system performance never achieved expectations at normal operating conditions, the pH attenuated control tests were not performed. The operating pH of the system was maintained at approximately 7.0 throughout the entire data collection and performance monitoring phases of the demonstration.

Table 5-13. Results of the Second Set of Laboratory Batch Tests

Test	Basic Salts	BSM B 800X	Water Used	Phenol Used	OD after 1 day	OD after 2 days	Phenol detected after 1 day	Phenol detected after 2 days	Precipitates Formed
1	site	site	site[a]	site	0.205	0.460	–	–	–
2	site	site	site[a]	site	0.220	0.460	–	–	–
3	site	site	site[b]	site	0.145	0.292	–	–	+
4	site	site	site[b]	site	0.149	0.310	–	–	+
5	site	site	site	site	0.019	0.028	+	+	+
6	site	site	site	site	0.017	0.028	+	+	+
7	2x site	site	site	site	0.015	0.032	+	+	+
8	2x site	site	site	site	0.002	0.030	+	+	+
9	site	site	DI	site	0.145	0.289	–	–	–
10	site	site	DI	site	0.151	0.292	–	–	–
11	site	site	DI[b]	site	0.148	0.305	–	–	–
12	site	site	DI[b]	site	0.149	0.290	–	–	–
13	site	2x site	site	site	0.015	0.088	+	+	+
14	site	2x site	site	site	0.025	0.030	+	+	+
15	site	site	site[c]	site	0.151	0.252	–	–	+
16	site	site	site[c]	site	0.135	0.232	–	–	+
17[d]	site	site	DI[b]	site	0.010	0.021	+	+	–
18[d]	site	site	DI[b]	site	0.010	0.020	+	+	–

(a) Potable site or DI laboratory water was softened with a cation exchange unit.
(b) Potable site or DI laboratory water was autoclaved.
(c) Potable site or DI laboratory water was filtered through GAC.
(d) For tests #17 and #18, the G-4 strain was autoclaved after addition to the reaction flasks.

5.1.6 System Shutdown

The final set of vapor samples for off-site analysis was collected on April 6, 1998 to check the performance of the system after the second re-inoculation. The results of this final sampling event are given in Tables 5-1 and 5-2. The results indicated that there was minor improvement in the degradation of 1,1-DCA, 1,2-*cis*-DCE, and toluene, however, the other contaminants of interest actually had higher concentrations in the CSTR effluent than the CSTR influent, demonstrating no contaminant removal.

The system was shut down and the data collection and performance monitoring phases of the project were completed on April 15, 1998. A sample of the biomass in the biofilter was taken and sent off-site for the final PLFA testing. The results of the PLFA analysis are given in Table 5-6. The demobilization and site restoration phase of the project was then started. Final demobilization from OU D was completed on April 30, 1998. The site was inspected by McClellan AFB personnel and the site checkout was completed on May 7, 1998.

5.1.7 Process Stream Characterization

Previous characterization sampling data of the SVE off-gas was provided to Battelle, so that the selection of the principal contaminants of concern could be made. During the demonstration, influent process gas stream VOCs were measured. Table 5-14 shows the concentration ranges of the VOCs measured using the U.S. EPA Method TO-14 analyses.

5.2 Vapor Removal Efficiency

As stated previously, the removal efficiency of the two-stage reactor system did not achieve process performance goals. The contaminant DREs target goal of 95% was only reached during initial startup and acclimation as a result of contaminant sorption to the biofilter medium. For most of the project demonstration, the removal efficiency of the total concentration of the contaminants of concern was approximately 60%. Individually, the contaminants were removed from the vapor at varying efficiencies. Table 5-15 lists the removal efficiencies calculated during data collection and performance monitoring for each contaminant of interest based on the off-site data generated. The removal efficiencies from the vapor phase were highest for 1,2-*cis*-DCE and toluene. The lowest removal efficiencies from the vapor phase were vinyl chloride and 1,1,1-TCA. Because the initial sampling event had removal efficiencies of over 95% for every contaminant of interest, it could be considered an outlying set of data. If the remaining data are averaged, the removal efficiencies for all of the contaminants of interest are below 63%, except for toluene (83%) and 1,2-*cis*-DCE (77%).

Table 5-14. Influent Process Gas VOC Concentrations for the Demonstration

Contaminant	Number of Analyses	Concentration Range (ppmv)	Concentration (ppmv)	
			Average	Std. Dev.
Freon® 12	6	ND to 0.68	0.303	0.284
Freon® 114	6	ND	ND	ND
Chloromethane	6	ND	ND	ND
Vinyl chloride	6	0.23 to 1.2	0.627	0.449
Bromomethane	6	ND	ND	ND
Chloroethane	6	ND	ND	ND
Freon® 11	6	ND to 0.064	0.011	0.024
1,1-Dichloroethene	6	3.2 to 7.5	4.600	2.288
Freon® 113	6	0.16 to 0.37	0.320	0.141
Methylene chloride	6	0.1 to 1.8	1.383	0.790
1,1-Dichloroethane	6	1.6 to 3.8	3.017	1.363
cis-1,2-Dichloroethene	6	1.0 to 3.8	2.067	1.289
Chloroform	6	ND	ND	ND
1,1,1-Trichloroethane	6	6.6 to 34	26.267	13.461
Carbon tetrachloride	6	ND	ND	ND
Benzene	6	ND to 0.15	0.025	0.057
1,2-Dichloroethane	6	ND to 0.2	0.100	0.088
Trichloroethene	6	6.4 to 18	14.900	6.902
1,2-Dichloropropane	6	ND	ND	ND
cis-1,3-Dichloropropene	6	ND	ND	ND
Toluene	6	4.1 to 9.3	7.567	3.309
trans-1,3-Dichloropropene	6	ND	ND	ND
1,1,2-Trichloroethane	6	ND to 0.18	0.068	0.080
Tetrachloroethene	6	3.5 to 22	17.750	9.295
Ethylene dibromide	6	ND	ND	ND
Chlorobenzene	6	0.10 to 0.42	0.298	0.151
Ethyl benzene	6	0.31 to 0.72	0.542	0.238
m,p-Xylene	6	1.4 to 3.2	2.383	1.045
o-Xylene	6	0.76 to 1.6	1.210	0.519
Styrene	6	ND	ND	ND
1,1,2,2-Tetrachloroethane	6	ND	ND	ND
1,3,5-Trimethylbenzene	6	1.8 to 3.0	2.150	0.928
1,2,4-Trimethylbenzene	6	5.8 to 10	7.300	3.229
1,3-Dichlorobenzene	6	0.92 to 1.8	1.200	0.540
1,4-Dichlorobenzene	6	2.4 to 4.4	3.017	1.326
Chlorotoluene	6	ND	ND	ND
1,2-Dichlorobenzene	6	8.8 to 24	15.467	7.624
1,2,4-Trichlorobenzene	6	ND to 0.31	0.113	0.139
Hexachlorobutadiene	6	ND	ND	ND
Propylene	6	ND to 0.17	0.028	0.064
1,3-Butadiene	6	ND	ND	ND
Acetone	6	0.29 to 2.6	1.465	0.927
Carbon disulfide	6	ND	ND	ND
2-Propanol	6	ND	ND	ND
trans-1,2-Dichloroethene	6	ND	ND	ND

Table 5-14. Influent Process Gas VOC Concentrations for the Demonstration

Contaminant	Number of Analyses	Concentration Range (ppmv)	Concentration (ppmv)	
			Average	Std. Dev.
Freon® 12	6	ND to 0.68	0.303	0.284
Freon® 114	6	ND	ND	ND
Chloromethane	6	ND	ND	ND
Vinyl chloride	6	0.23 to 1.2	0.627	0.449
Bromomethane	6	ND	ND	ND
Chloroethane	6	ND	ND	ND
Freon® 11	6	ND to 0.064	0.011	0.024
1,1-Dichloroethene	6	3.2 to 7.5	4.600	2.288
Freon® 113	6	0.16 to 0.37	0.320	0.141
Methylene chloride	6	0.1 to 1.8	1.383	0.790
1,1-Dichloroethane	6	1.6 to 3.8	3.017	1.363
cis-1,2-Dichloroethene	6	1.0 to 3.8	2.067	1.289
Chloroform	6	ND	ND	ND
1,1,1-Trichloroethane	6	6.6 to 34	26.267	13.461
Carbon tetrachloride	6	ND	ND	ND
Benzene	6	ND to 0.15	0.025	0.057
1,2-Dichloroethane	6	ND to 0.2	0.100	0.088
Trichloroethene	6	6.4 to 18	14.900	6.902
1,2-Dichloropropane	6	ND	ND	ND
cis-1,3-Dichloropropene	6	ND	ND	ND
Toluene	6	4.1 to 9.3	7.567	3.309
trans-1,3-Dichloropropene	6	ND	ND	ND
1,1,2-Trichloroethane	6	ND to 0.18	0.068	0.080
Tetrachloroethene	6	3.5 to 22	17.750	9.295
Ethylene dibromide	6	ND	ND	ND
Chlorobenzene	6	0.10 to 0.42	0.298	0.151
Ethyl benzene	6	0.31 to 0.72	0.542	0.238
m,p-Xylene	6	1.4 to 3.2	2.383	1.045
o-Xylene	6	0.76 to 1.6	1.210	0.519
Styrene	6	ND	ND	ND
1,1,2,2-Tetrachloroethane	6	ND	ND	ND
1,3,5-Trimethylbenzene	6	1.8 to 3.0	2.150	0.928
1,2,4-Trimethylbenzene	6	5.8 to 10	7.300	3.229
1,3-Dichlorobenzene	6	0.92 to 1.8	1.200	0.540
1,4-Dichlorobenzene	6	2.4 to 4.4	3.017	1.326
Chlorotoluene	6	ND	ND	ND
1,2-Dichlorobenzene	6	8.8 to 24	15.467	7.624
1,2,4-Trichlorobenzene	6	ND to 0.31	0.113	0.139
Hexachlorobutadiene	6	ND	ND	ND
Propylene	6	ND to 0.17	0.028	0.064
1,3-Butadiene	6	ND	ND	ND
Acetone	6	0.29 to 2.6	1.465	0.927
Carbon disulfide	6	ND	ND	ND
2-Propanol	6	ND	ND	ND
trans-1,2-Dichloroethene	6	ND	ND	ND

Table 5-14. Influent Process Gas VOC Concentrations for the Demonstration (Continued)

Contaminant	Number of Analyses	Concentration Range (ppmv)	Concentration (ppmv)	
			Average	Std. Dev.
Vinyl Acetate	6	ND	ND	ND
Chloroprene	6	ND	ND	ND
Metyl ethyl ketone	6	ND	ND	ND
Hexane	6	ND	ND	ND
Tetrahydrofuran	6	ND	ND	ND
Cyclohexane	6	ND	ND	ND
1,4-Dioxane	6	ND to 0.48	0.117	0.187
Bromodichloromethane	6	ND	ND	ND
4-Methyl-2-pentanone	6	ND	ND	ND
2-Hexanone	6	ND	ND	ND
Dibromochloromethane	6	ND	ND	ND
Bromoform	6	ND	ND	ND
4-Ethyltoluene	6	2.4 to 4.0	3.150	1.349
Ethanol	6	ND	ND	ND
Methyl-*tert*-butyl ether	6	ND	ND	ND
Heptane	6	ND to 0.19	0.032	0.072

ND = Not detected.

Table 5-15. Removal Efficiencies from the Vapor Stream

Sample Date	Removal Efficiency (%)						
	VC	1,1-DCA	1,2-*cis*-DCE	1,1,1-TCA	TCE	PCE	Toluene
12/23/97	99.80	99.80	99.92	99.77	99.76	99.71	99.92
01/13/98	43.48	46.67	76.00	43.75	50.59	47.62	85.23
01/30/98	62.08	63.16	63.08	64.71	63.53	65.00	68.82
03/02/98	58.06	56.25	83.64	53.57	58.57	54.76	87.89
04/06/98	58.33	80.53	86.05	64.44	77.65	75.26	91.50
Average	64.35	69.28	81.74	65.25	70.02	68.47	86.67

5.3 Process Flow Efficiency

Process upsets were frequent during this project demonstration because of the numerous shutdowns of the SVE system that supplied the contaminated air stream, and because of the two re-inoculation events. The total reactor system operational time was approximately 60 to 65% of the total demonstration time. The CSTR was re-inoculated with the G-4 media on two separate occasions. Because after each re-inoculation the CSTR had to be operated in batch mode, an additional 18 days of operational time was used in order to grow the fresh bacteria in the CSTR.

Approximately 504,000 ft^3 of contaminated vapor was treated by the two-stage reactor system. Additionally, approximately 1,600 gallons of wastewater was generated during the project demonstration and was disposed of as waste by McClellan AFB personnel.

Taking installation tasks (3 days), G-4 bacteria growth periods (30 days), and shutdown and demobilization tasks (15 days) into account, contaminated air was fed to the two-stage reactor system 92 out of a possible 150 days. The most effective treatment of the contaminated off-gas from the SVE took place during the initial period of the project from December 3 through December 23, 1997. After this period, the SVE shutdowns became more frequent. Table 5-16 provides the project demonstration timeline and events. Only extended periods of SVE shut-downs are indicated in Table 5-16. Shorter SVE shutdowns of approximately 2 to 8 hours occurred during the demonstration, but are not indicated in the table.

Table 5-16. Project Demonstration Timeline

Date	Event	Project Demonstration Time (days)	SVE Downtime (days)	System Operational Time (days)
11/13/97	System installation	0	0	0
11/16/97	System startup and shakedown	3	0	0
12/03/97	Abiotic testing phase	20	0	17
12/04/98	Reactor inoculation	21	0	18
12/17/98	Switched from batch to continuous operation	34	0	31
12/17/98	Data collection and testing phase	34	0	31
12/27/97	SVE shutdown	44	0	41
01/08/98	SVE startup	56	12	41
01/13/98	Performance monitoring phase	61	12	46
01/19/98	SVE shutdown	67	12	52
01/26/98	SVE startup	74	19	52
02/04/98	SVE shutdown	83	19	61
02/10/98	SVE startup	89	25	61
02/11/98	1st re-inoculation	90	25	62
02/19/98	Switched from batch to continuous operation	98	25	70
03/03/98	SVE shutdown	110	25	82
03/05/98	SVE startup	112	28	82
03/11/98	SVE shutdown	118	28	88
03/12/98	SVE startup	119	30	88
03/13/98	2nd re-inoculation	120	30	89
03/16/98	SVE shutdown	123	30	92
03/21/98	Switched from batch to continuous operation	128	35	92
03/22/98	SVE startup	129	36	92
04/01/98	SVE shutdown	139	36	102
04/05/98	SVE startup	143	40	102
04/15/98	System shutdown and demobilization phase	153	40	112
04/30/98	Demobilization complete	168	40	112
05/07/98	Site checkout complete	175	40	112

6.0 Other Technology Issues

This section describes all applicable or relevant regulatory requirements related to the activities discussed in this report. These requirements included the acquisition of any necessary permits to complete the field demonstration and the compliance with any regulations, which applied to the demonstration. The permitting and compliance issues are described below.

6.1 Environmental Regulatory Requirements

No additional permits were required for on-site operations. However, all operations complied with any applicable federal, state, and local regulations for which permits normally would be required. Operations that were subject to such regulations are discussed in Sections 6.1.1 through 6.1.5. Furthermore, the exemption from a specific permitting process was not applicable to any off-site operations, including the transport of materials or products to the site or off site. Any activities that occurred off site were subject to the appropriate permitting procedures.

6.1.1 Hazardous Materials Storage

No permits were required for hazardous materials storage. The contractor was required to obtain approval from Sacramento Air Logistics Center, Environmental Compliance Branch (SM-ALC/EMPC), before bringing any hazardous materials to the site.

6.1.2 Air Discharge

Air discharge permits (under Title V for the Clean Air Act [CAA]) were not required. The process gas stream was passed through two carbon canisters, in series, and was monitored daily using a hand-held PID and weekly using the on-site GC to determine contaminant concentrations in the vapor effluent from the canisters. U.S. EPA Method TO-14 analyses at an off-site CLP laboratory were used to monitor the vapor effluent from the system. Discharge concentrations were maintained above the 95% regulated organic compound (ROC) removal requirements established by SMAQMD.

6.1.3 Wastewater Discharge

The Air Force was responsible for wastewater discharge. The contractor ensured that the wastewater was properly transferred to the wastewater holding tank. Contaminant concentrations in the wastewater were identified by the off-site analyses performed during the demonstration, before McClellan AFB discharged the wastewater.

6.1.4 Waste Storage, Treatment, and Disposal

Only satellite accumulation of hazardous waste materials was permitted on the job site. Transport of hazardous waste from the job site and disposal were handled under contract to McClellan AFB. Industrial waste accumulation and disposal were not permitted on the job site. No additional permits were required for work at McClellan AFB. Work at off-site locations was subject to the individual facility's regulatory and permitting requirements.

6.1.5 General Operations

No additional permits were required.

6.2 Regulatory Compliance

In addition to fulfilling the requirements cited in Section 6.1, the implementation and operation phases of the technology demonstration were required to comply with several other Federal, state, and local regulations, including but not limited to the following:

6.2.1 Comprehensive Environmental Response, Compensation, and Liability Act

McClellan AFB has been listed on the National Priorities List under Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). The requirements of CERCLA and other applicable or relevant and appropriate requirements (ARARs) were adhered to throughout the study.

6.2.2 Resource Conservation and Recovery Act

The Resource Conservation and Recovery Act (RCRA) as amended (42 United States Code [USC] §901 et seq.) was adhered to for any hazardous waste generated that would not be considered investigation-derived waste (IDW) under the requirements of CERCLA.

6.2.3 Clean Water Act and Clean Air Act

Regulation under the Clean Water Act (CWA) as amended (Title 42 USC §401 et seq.) was adhered to in all operations at McClellan AFB. The operations did not result in liquid emissions to surface water or groundwater.

Regulations under the CAA as amended (Title 42 USC §401 et seq.) was adhered to in all operations at McClellan AFB. The operations did not result in gaseous, particulate, or thermal emissions to the atmosphere.

6.2.4 Safe Drinking Water Act

All operations conducted at McClellan AFB were conducted so as to ensure the quality of drinking water as regulated under the Safe Drinking Water Act (SDWA) as amended (42 USC §300f et seq.). The operations did not result in discharges to surface water or groundwater.

6.2.5 Toxic Substances Control Act

Not applicable for this project.

6.2.6 Mixed Waste Regulations

Not applicable for this project.

6.2.7 Federal Insecticide, Fungicide, and Rodenticide Act

Not applicable for this project.

6.2.8 Occupational Safety and Health Act

U.S. Occupational Safety and Health Administration standard work practices and safety standards under the Occupational Safety and Health Act (OSHA) as amended (29 USC §51 et seq.) were adhered to throughout the duration of the project. For work not on U.S. Government property, state OSHA program requirements were followed. All work was conducted in accordance with the McClellan AFB Spill Prevention, Control, and Countermeasures Plan (SPCCP) (McClellan AFB, 1996), and a project-specific health and safety plan (Battelle and Envirogen, Inc., 1997b).

6.2.9 State and Local Regulations

6.2.9.1 State of California Regulations

All pertinent state requirements were observed (i.e., state implementation of RCRA).

6.2.9.2 SMAQMD Regulations

Reactor off-gas effluent was passed through a two-stage GAC filter unit. The GAC off-gas was monitored to ensure that the SMAQMD 95% DRE requirement was achieved throughout the study.

6.2.9.3 Air Force Regulations

Air Force regulations were followed. McClellan AFB was notified of all upcoming and ongoing activities throughout the duration of the project. All work was done in accordance with the McClellan AFB SPCCP (McClellan AFB, 1996).

6.2.9.4 DoD Directives

DoD regulations implementing Federal and state regulations were followed.

6.3 Personnel Health and Safety

Personnel health and safety was a priority concern during this demonstration. Section 9.0 of the work plan for this project (Battelle and Envirogen, Inc., 1997b) details the safety issues and methods done to ensure the health and safety of the on-site and off-site personnel who worked on this project.

6.4 Community Acceptance

Because the two-stage bioreactor process did not achieve the targeted DREs goal of 95%, it is unlikely that the public would be willing to accept this remedial alternative until it can meet CAA requirements.

7.0 Cost Analysis

During the demonstration of the two-stage reactor system, project costs were monitored to provide information necessary to scale-up costs for a full-scale implementation cost estimate. Because the technology was unsuccessful at destroying the VOCs in the off-gas, full-scale implementation of this technology at McClellan AFB would be impractical with respect to effectiveness for remediating chlorinated VOCs and for cost. For this reason, and with Air Force concurrence, full-scale implementation costs were not calculated. However, Section 7.1 presents the actual costs expended during the pilot-scale technology implementation at McClellan AFB.

7.1 Basis of Cost Analysis

The basis of the cost analysis was the incurred costs during the project demonstration. Table 7-1 lists the costs incurred for the two-stage reactor system demonstration. The reactor system equipment costs were calculated by amortizing the system equipment on a per usage basis. The chemical costs were for the calibration gases, phenol, ammonium nitrate, mono-potassium phosphate, *P. cepacia* strain G-4 bacteria, and caustic sodium hydroxide used during the project. The biomass inoculum and the carbon canisters used are included as a separate project cost from the chemical usage cost. The utilities costs for water, telephone, and electricity were based on local usage rates. The disposal costs shown in Table 7-1 were based on estimated costs incurred by the Air Force to remove the wastewater and solids generated during the demonstration.

7.2 Cost Categories

Not applicable for this project.

Table 7-1. Field Pilot Demonstration Costs

Cost Component	Demonstration Cost[a]
Equipment Acquisition	
Equipment Capital Cost	$82,731
Monthly Equipment Rental Cost	$1,379
Equipment Preparation (12 weeks)	
Materials	
Consumables and parts	$1,009
Contractor (Conceptual process flow diagrams)	$3,363
Labor	
Supervisor	$10,164
Project manager	$9,432
Field engineers	$10,365
Engineering and administrative	$3,883
Shipping charges	$372
System Setup (5 weeks)	
Materials	
Inoculum and inoculum preparation	$1,550
Consumables and parts	$1,940
Equipment delivery and shipping	$8,125
Labor	
Supervisor	$1,739
Project manager	$8,140
Field engineer	$15,635
Engineering and administrative	$127
On-site contractors	$798
Travel	$7,274
Shipping charges	$272
System Operation (18 weeks)	
Materials	
Activated carbon	$1,540
Phenol	$1,010
Caustic solution and nutrients	$406
Antifoam	$262
Field analytical supplies	$2,010
Consumables and parts	$1,271
Off-site analyses	$18,045
Labor	
Supervisor	$5,652
Project manager	$12,528
Field engineer	$34,475
Engineering and administrative	$2,120
On-site contractors	$704
Travel	$1,008
Shipping charges	$1,109

Table 7-1. Field Pilot Demonstration Costs (Continued)

Cost Component	Demonstration Cost[a]
Demobilization and Decommisioning (1 week)	
Materials	
Consumables and parts	$221
Equipment removal and shipping	$4,590
Labor	
Project manager	$1,789
Field engineer	$1,690
Engineering and administrative	$73
Travel	$1,018
TOTAL PROJECT COSTS	$181,225

(a) Costs include fringe benefits, overhead, and general and administrative (G&A). Does not include fee.

7.3 Results of Cost Analysis

The total cost without fee to perform the pilot-scale demonstration was $181,225. This does not include the reporting costs incurred during the project. The estimated costs for the full-scale implementation of the two-stage reactor system were not performed due to the unsuccessful removal of chlorinated VOCs from the contaminated air stream.

8.0 Recommendations

For reasons that could not be determined during this study, the CSTR DREs were much lower than expected. Total DREs ranged from 43 to 73% in the biofilter and from zero to 15% in the CSTR. Total system DREs ranged from 51 to 74%, and did not meet the target DRE of 95%. Because of these low DREs, the two-stage reactor system design demonstrated is not recommended for treatment of the SVE off-gas at McClellan AFB.

9.0 Conclusions

For reasons that could not be determined during this study, the CSTR DREs were much lower than expected and did not meet the performance criterion of 95% contaminant destruction. Total system DREs for the two-stage reactor process ranged from 51 to 74%, which still did not achieve the 95% target DRE goal. The first stage biofilter performed as expected with high DREs for nonchlorinated compounds, as shown for toluene. However, DREs for chlorinated compounds were higher than expected in the biofilter. The removal mechanism for the chlorinated compounds remains unclear, but could be attributed in part to sorption or to biological transformation. In spite of the higher removal efficiencies, the biofilter did not meet the target DREs of 95%.

9.1 Cost and Performance

During the demonstration of the two-stage reactor process, project costs were monitored to provide the necessary information to scale up costs for the full-scale implementation cost estimate. However, since the two-stage reactor system was not successful in removing VOCs from the SVE off-gas, the cost estimate for the full-scale implementation of the process was not performed. Refer to Section 7.0 for the pilot-scale demonstration cost information.

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Appendix A

Appendix A. Phospholipid Fatty Acid Analysis Report

Two sets of samples (BFI-S-N148, BFI-S-N149QAD and BFI-S-N498, BFI-S-N498QAD) were extracted from the biofiltration reactor and analyzed for changes in phospholipid fatty acid (PLFA) content. The first set of samples was extracted from the biofilter in duplicate and sent for analysis before the project began (12/17/97). The second set of samples (in duplicate) was sent for analysis after the project ended (4/15/98). Using the PLFA as indicators, the samples were analyzed for total biomass content, community structure, phase of growth (logarithmic or stationary), and physiology (stress).

X.1 Biomass

The biomass measurement is the quantity of PLFA detected in a sample. Phospholipids are part of the intact cell membranes; thus, the biomass is a measure of viable or potentially viable cells. As the cell ruptures, phospholipids are attacked by enzymes resulting in a diglyceride fatty acid (DG) molecule representative of recently dead or lysed microorganisms.

The cell equivalent value is calculated from experiments with typical bacteria isolated from soil and water. This value is based on 2.0×10^{12} cells per gram dry weight of cells and 10^{8} picomoles of phospholipid/gram dry weight of cells. This gives between 1.4×10^{4} and 4.0×10^{4} cells per picomole (10^{-12}) of PLFA. The number of cells/gram of dry weight may vary depending on the environmental conditions from which the microorganisms were recovered.

The first set of samples contained a substantial amount of biomass and a procaryotic to eucaryotic ratio of three to one. The relative percent difference between the duplicate samples for the number of cells per gram of dry soil was 23 %. This difference is attributable to the heterogeneity of the solid matrix extracted and sampled (Table 1 and Figure 1). The second set of samples demonstrated a 20 % increase in biomass over the course of the experiment and an increase in the procaryotic to eucaryotic ratio up to five to one (Table 2 and Figure 2). The increase in biomass was expected as the community developed within the reactor as contaminants were consumed. The increase in the procaryotic to eucaryotic ratio demonstrates that the growth of the procaryotic microbes exceeded the predation carried out by the eucaryotic organisms.

TABLE 1. Biomass Calculations.

Sample	***BF-I-S-N148***	***BF-I-S-N149-QAD***
picomoles total PLFA/g dry solid	113,169	146,011
Cells/g dry solid	2.26E+09	2.92E+09
picomoles procaryote PLFA	85,771	112,501
picomoles eucaryote PLFA	27,432	33,495
ratio procaryote/eucaryote	3	3

TABLE 2. Biomass Calculations:

Sample Name	*BFI-S-N498*	*BFI-S-N498QAD*
picomoles total PLFA/g dry soil	178,293	177,726
Cells/g dry soil	3.57E+09	3.55E+09
picomoles procaryote PLFA	150,248	149,823
picomoles eucaryote PLFA	28,028	27,885
ratio procaryote/eucaryote	5	5

FIGURE 1. Biomass content in the samples taken 12/17/97.

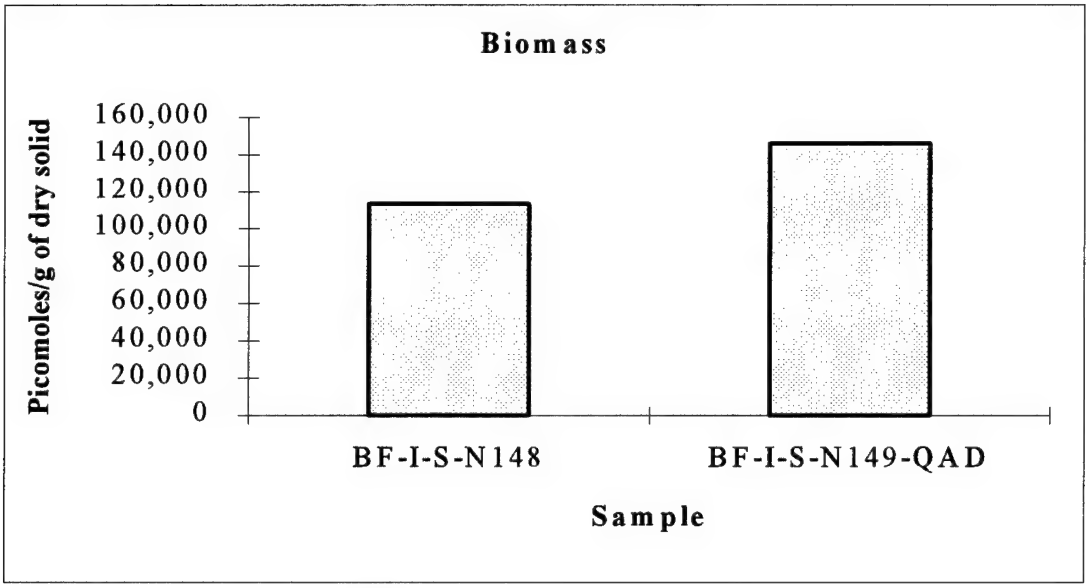
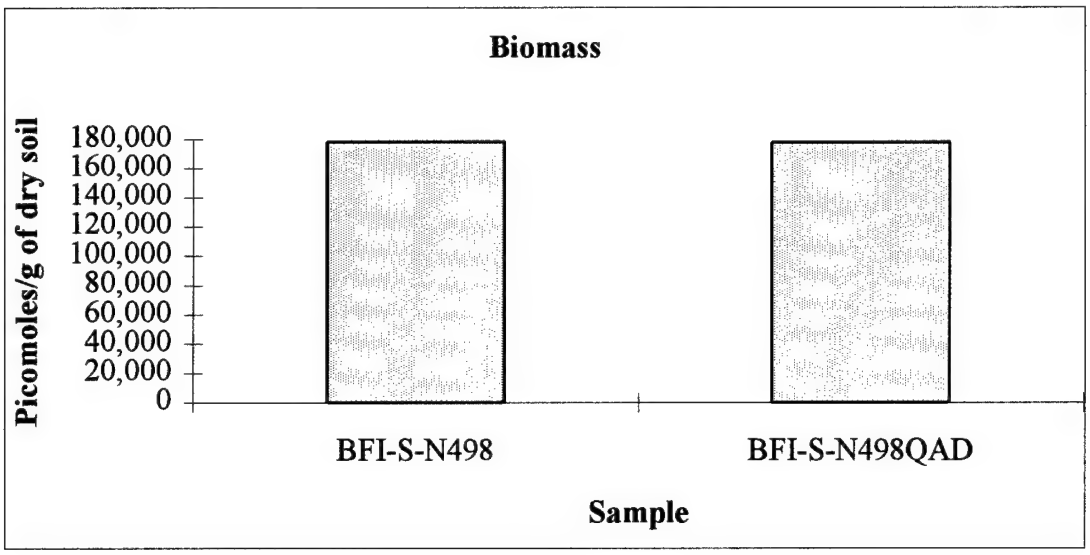


FIGURE 2. Biomass content in the samples taken 4/15/98.



X.2 Community Structure

Community structure data is defined by different classes of fatty acids that indicate the presence of certain microbial types. One note of caution is that the different classes of acids are presented as a percent of the total community. Direct comparison of these percentages between two different sets of samples are valid only for determining shifts in the types of microbes that dominate the community as a whole. The percentages presented simply normalize the data. Thus, the actual number of microbes per class of fatty acid will depend on both the percentage and the total number of microbes within each sample.

The first set of samples was primarily composed of monoenoic PLFA (Monos) (~39.1 %, Figure 3). The relative percentage of monoenoic PLFA did not change over the course of the experiment (BFI-S-N498 (39.2%) and BFI-S-N498QAD (40.6%) (Figure 4). Generally, monoenoic PLFA are found in Gram negative bacteria, which are fast growing, utilize many carbon sources, and adapt quickly to a variety of environments. The monoenoic biomarker for the sulfate reducing bacteria *Desulfubulbus*, (17:1ω6c) was detected in both samples of the first set (0.4%) and in one of the samples (BFI-S-N498) of the second set (0.2%). A decline in this value would indicate that anaerobic conditions were not prevalent within the biofilter media.

FIGURE 3. Diversity of the microbial communities in the samples taken 12/17/97.

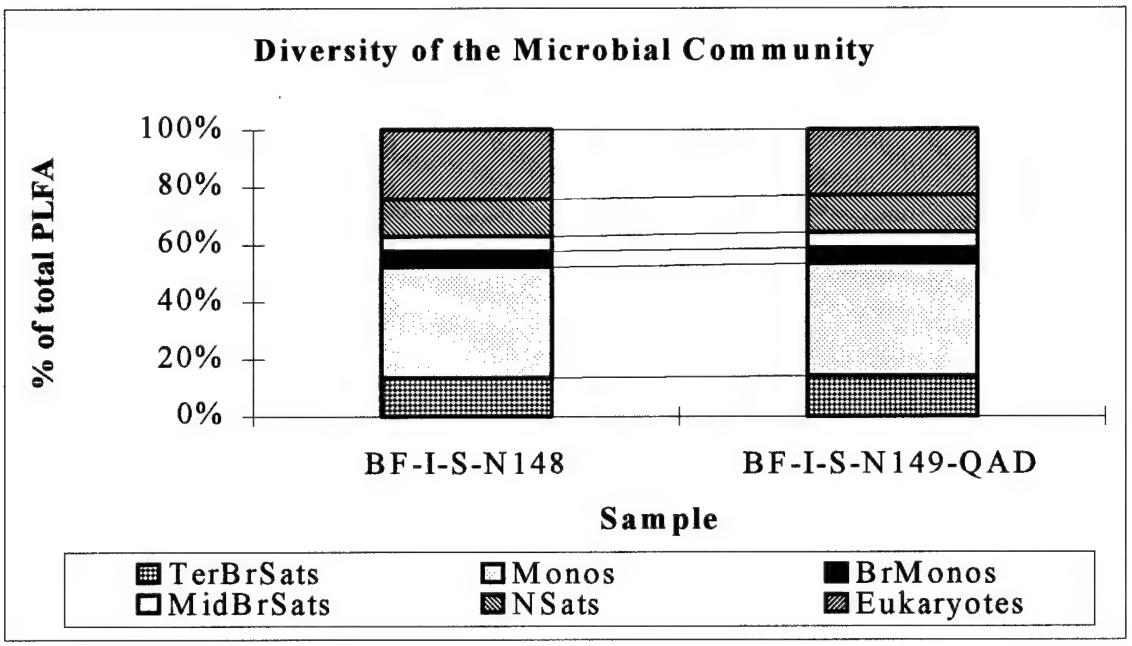
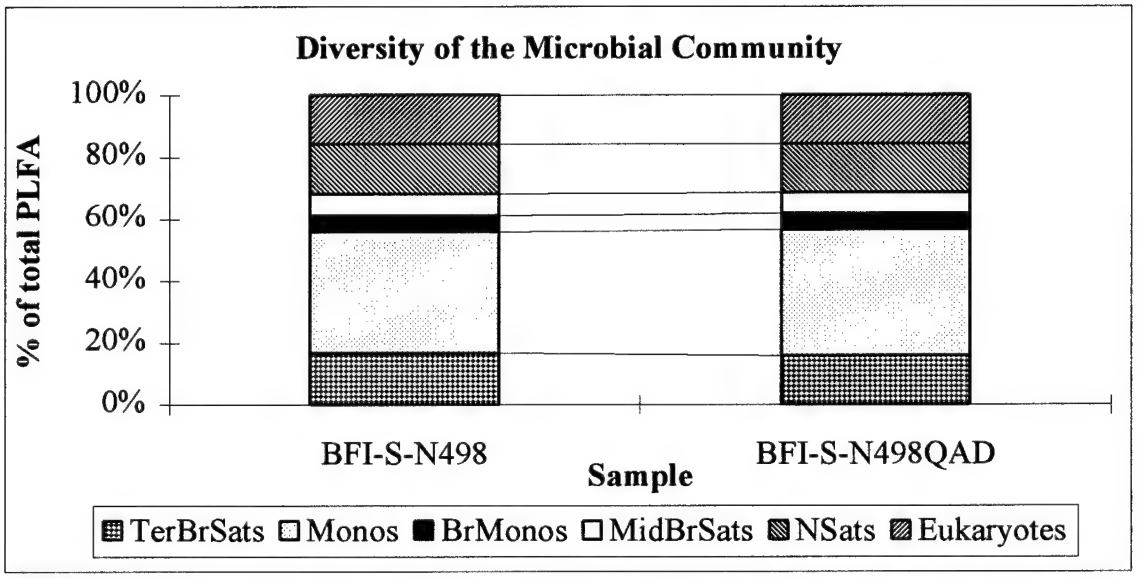


FIGURE 4. Diversity of the microbial communities in the samples taken 4/15/98.



Terminally branched saturated PLFA are representative of Gram positive bacteria but may also be found in the cell membranes of many sulfate reducing bacteria. Generally, Gram positive bacteria are slower growing than Gram negative bacteria, more resilient, and are capable of degrading more complex compounds. Terminally branched saturated PLFA (TerBrSats) were detected in both samples (~13.7%) of the first set. A small percentage increase was seen over the course of the experiment as demonstrated by the results of the second set of samples (BFI-S-N498 (16.7%) and BFI-S-N498QAD (15.9%)).

Branched monoenoic PLFA (BrMonos) were detected in both samples of the first sample set (~5.4%) as well as in the samples of the second set (BFI-S-N498 (5.3%) and BFI-S-N498QAD (5.2%)). Branched monoenoic PLFA are commonly found in the cell membranes of obligate anaerobes such as sulfate or iron reducing bacteria. Little change occurred over the duration between sample sets, possibly indicating that the development of anaerobic areas within the biofilter media was limited.

An increase in mid-chain branched saturated PLFA (MidBrSats) was detected between the first and second set of samples. The first set contained an average of approximately 5.4% while the second set averaged 6.8%. Mid-chain branched saturated PLFA are common in *Actinomycete* spp., sulfate reducing bacteria and certain Gram positive bacteria. The biomarker 10me16:0, found in the anaerobic sulfate or iron reducing bacteria *Desulfobacter* was detected in both samples (~3.6%) of the first set. A decline in this percentage (~2.7%) was seen for the second set of samples. The biomarker 10me18:0, found in *Actinomycete* spp., was detected in both samples (~0.7%) of the first set. An increase in this biomarker was seen for the second set up to 1.5 %. *Actinomycetes* are of particular importance because they decompose complex or resistant organic compounds and can function at relatively high temperature levels.

Eucaryote PLFA were detected in both samples of the first set (~23.6%) and a decline was noticed in the relative percent within the second set of samples (15.7%). Eucaryote PLFA are found in organisms such as fungi, protozoa, algae, higher plants and animals. There are several biomarkers for different microeucaryotic organisms. The biomarker 18:2ω6 which is prominent in fungi, but is also found in algae, protozoa, higher plants and animals was detected in both samples (~9.3%) of the first set. A decline in this biomarker was seen for the second set of samples (~3.8%). Fungi are aerobic, grow over a wide pH range, are versatile in adapting to hostile environments and are particularly adapted to the decomposition of complex organic compounds. The biomarker 18:3ω3, found in fungi, algae, and higher plants, was detected in the first and second set of samples (0.6%). The biomarkers found in protozoa (20:4ω6) (~1.3%) and diatoms and higher plants (20:5ω3)(~0.6%) were detected in first set of samples. Minimal change was seen in the second set of samples with the protozoa (20:4ω6) (~1.0%) and diatoms and higher plants (20:5ω3) (0.4%). The biomarker 18:3ω6 indicative of fungi was also detected in both sets of samples (~0.7% and 0.6%, respectively). The eucaryote profiles for both sets of samples contained a relatively large proportion of the fatty acid 18:1ω9c, which is a precursor for certain eucaryote PLFA and due to the presence of biomarkers indicative of microeucaryotes, is included in the PLFA profile. However, this fatty acid is also detected in the Gram negative bacteria and its inclusion in the eucaryote profile may lead to a slight overestimation of the level of microeucaryotes and an underestimation of the relative proportion of Gram negative bacteria in these samples.

X.3 Metabolic Status

X.3.1 Growth Phase

In Gram negative bacteria, the monoenoics (16:1ω7c & 18:1ω7c) are converted to cyclopropyl fatty acids (cy17:0 & cy19:0) as microbes move from a log to a stationary phase of growth (i.e. slowing of growth). This change is expressed in the two ratios cy17:0/16:1ω7c and cy19:0/18:1ω7c. The ratios vary from organism to organism or environment to environment but usually will fall within the range of 0.05 (log phase) to 2.5 (stationary phase). When the ratios are summed the range is from 0.1 to 5.0. An increase in cyclopropyl formation has also been associated with anaerobic metabolism. The ratio is inversely proportional to the turnover rate, i.e., a lower ratio infers a higher turnover rate.

The Gram negative communities in both sets of samples were demonstrated to be in the stationary phase of growth (Figure 5 and 6).

FIGURE 5. Growth phase of the Gram negative communities for samples taken 12/17/97.

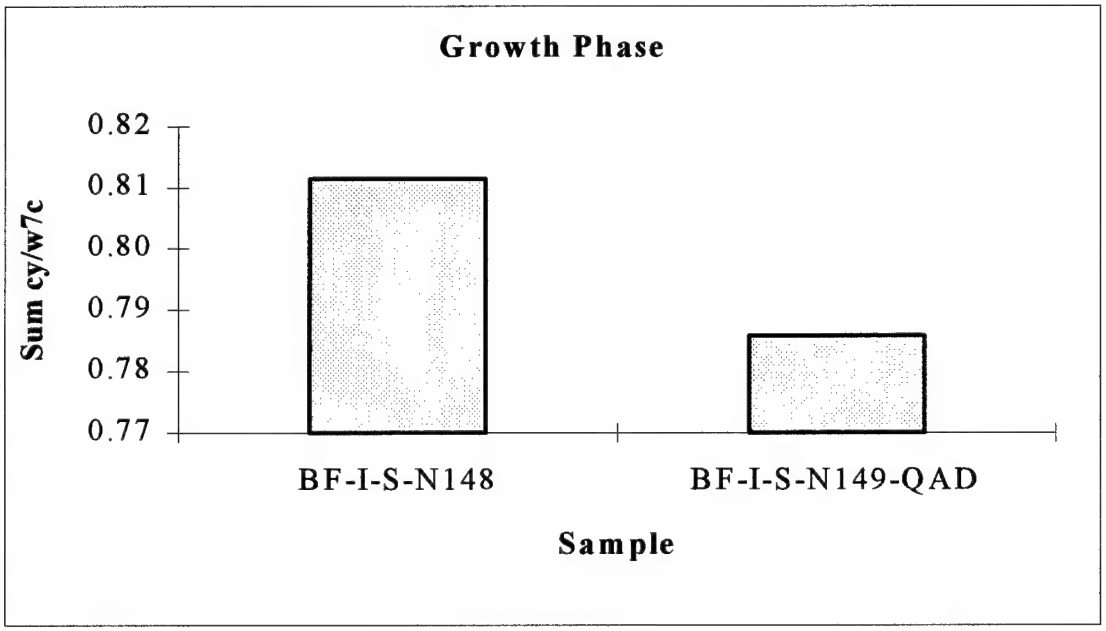
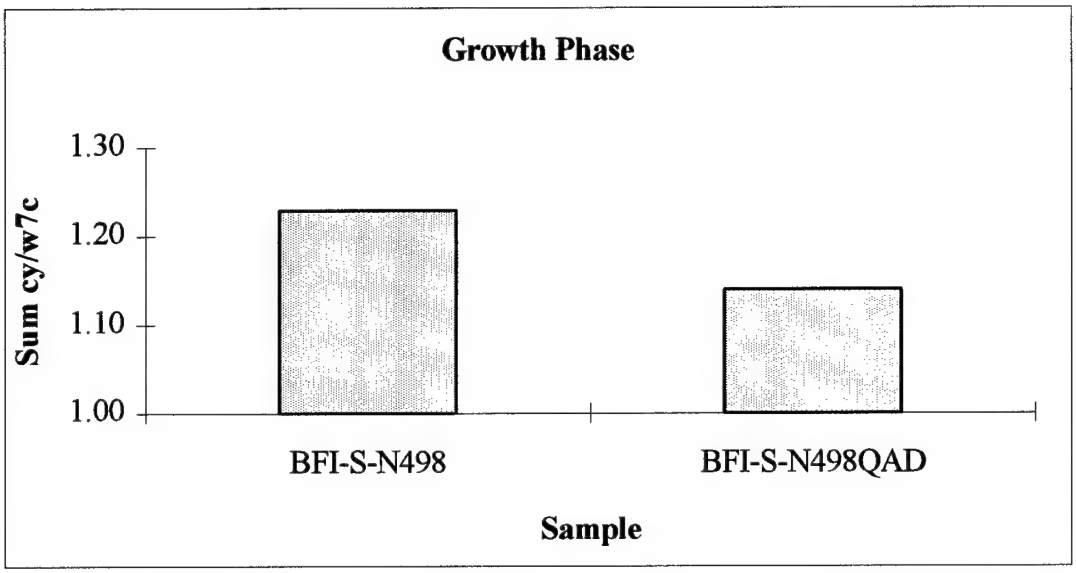


FIGURE 6. Growth phase of the Gram negative communities for samples taken 4/15/98.



X.3.2 Stress Factor

Gram negative bacteria also generate *trans* fatty acids to minimize the permeability of their cell membranes as protection against changes in the environment such as toxicity or starvation. For example, the bacteria make w7t fatty acids in the presence of toxic pollutants like phenol. Ratios (16:1ω7t/16:1ω7c and 18:1ω7t/18:1ω7c) greater than 0.1 have been shown to indicate the effects of starvation on bacterial isolates. The range is generally between 0.05 (healthy) to 0.3 (starved), or 0.1 (healthy) to 0.6 (starved) when the two are summed.

The Gram negative communities in the first sample set were not experiencing environmental stress from either toxicity or starvation (FIGURE 7). However, the Gram negative communities of the second sample set were adapting to low levels of environmental stress from either toxicity or starvation (FIGURE 8).

FIGURE 7. Level of environmental stress in the Gram negative communities for samples taken 12/17/97.

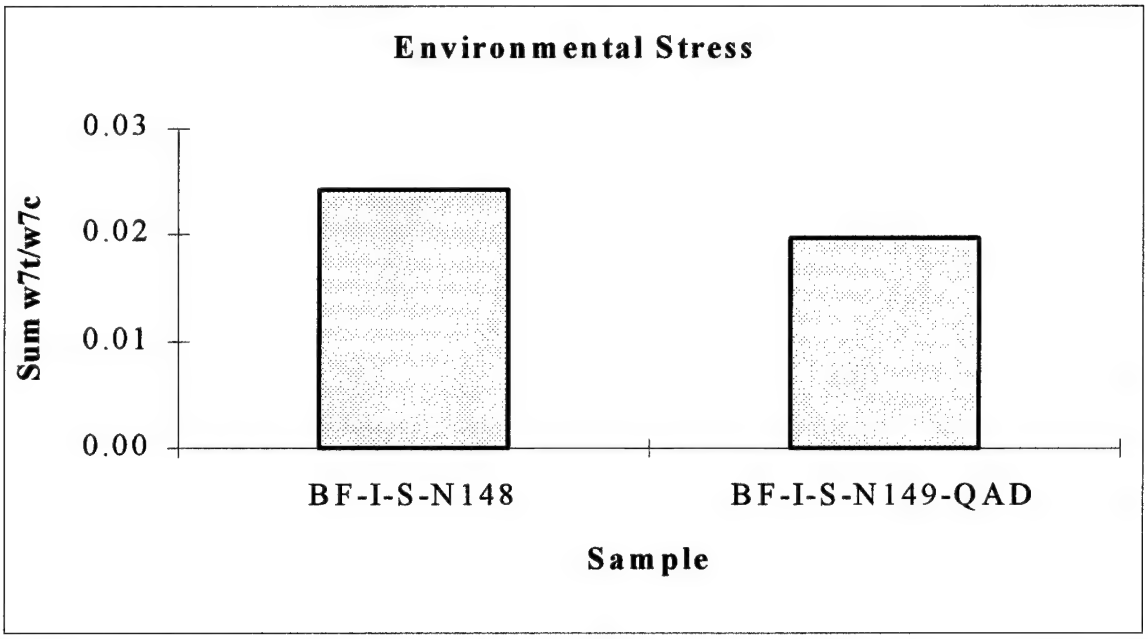
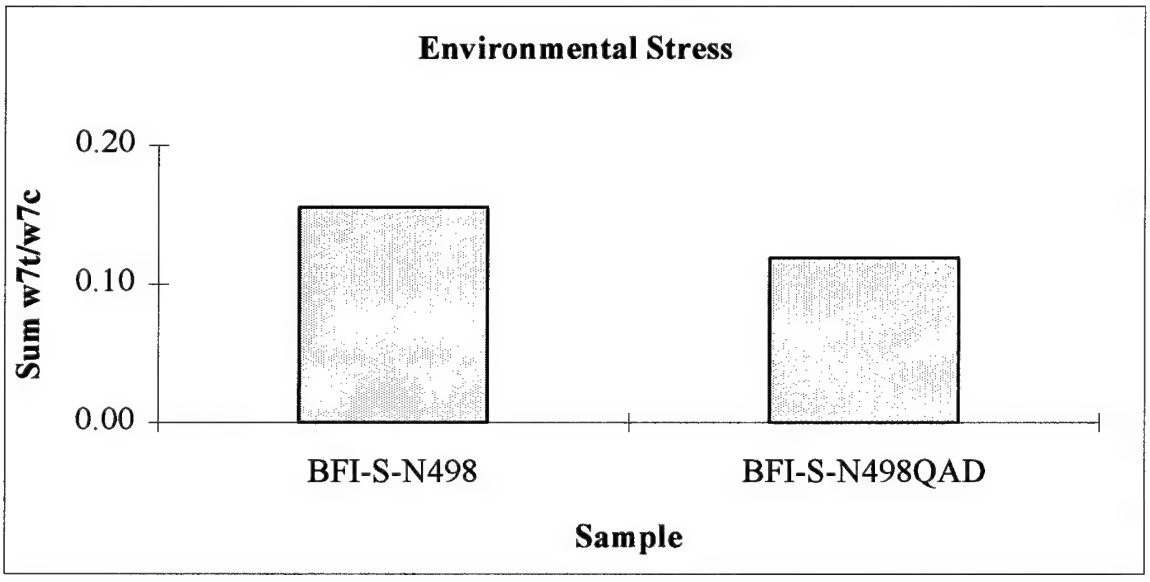


FIGURE 8. Level of environmental stress in the Gram negative communities for samples taken 4/15/98.



X.4 Conclusions

Two sets of biofilter compost samples were analyzed before the experiment commenced and after the completion of the project. Using PLFA analysis, changes in biomass concentration, community structure, phase of growth, and physiology were measured. Both sets of samples contained a substantial amount of biomass, with a 20 % increase occurring between the two

sampling events. Both sample sets contained very similar microbial communities, primarily composed of Gram negative bacteria. However, the proportion of the gram negative microbes (procaryotes) to the eucaryotes increased over the course of the experiment. Procaryotic organisms are generally responsible for the degradation of the contaminants entering the biofilter, thus their numbers predictably increased. Because the eucaryotic microbes did not increase proportionally with the procaryotes, it possibly may be inferred that the rate of predation was hindered. Had the experiment continued, this trend could have lead to an interesting condition where the procaryotes would have solely dominated the media within the reactor. Generally, such uncontrolled increasing biomass is not beneficial to the system.

The Gram negative communities in both sets of samples were in the stationary phase of growth while the PLFA results of the microbes in the second set demonstrated that some stress within the community was evident. This stress may have been related to the constant system interruption because the soil vapor extraction system repeatedly failed. Such a failure caused the microbes to exist without a food source during these shutdowns, possibly creating starvation conditions.

Evidence indicative of Gram positive bacteria, sulfate reducing bacteria, and microeucaryotes such as fungi, algae, protozoa, higher plants and animals were detected in both sample sets to varying degrees. The presence of numerous types of microbes within the media indicates a healthy ecosystem that should be beneficial for contaminant removal.

Appendix B

Appendix B. Quality Assurance and Control (QA/QC) Report

Acceptable and verifiable data were generated during the course of the experiment. A sufficient number of critical measurements for proper data evaluation were acquired for both the air and water analyses. However, because of the constant shutdowns with the soil vapor extraction system, a limited number of samples for off-site air and water analysis were obtained. Hence, the total number of quality control samples actually collected is less than the number anticipated in the work implementation plan.

Off-Site Air Sampling and Analysis Protocol

Air analyses were performed by Air Toxics LTD. (Sacramento, CA) using Environmental Protection Agency Methods TO-14 (Volatile Organics) and TO-12 (Total Non-Methane Hydrocarbons). Six-liter Summa® canisters were used for sample gas collection. These canisters were shipped to the site from the laboratory one day prior to the commencement of the sampling event. Prior to shipping, Air Toxics LTD. would perform the necessary preparation and cleaning of the canisters to ensure quality.

The preparation of the Summa® canisters involved first purging for one hour with humidified ultrapure air. Following this, the canisters were purged using a roughing pump and prepared for mounting on a high vacuum heating system (thermal clean-up and final evacuation). Three canisters at a time were heated to 125 °C for a minimum of 1 hour. A vacuum pump was used to evacuate the air in the canisters to less than or equal to 10 mTorr. The valves on the canisters were closed and a brass plug was placed on each canister. Ten percent of the canisters cleaned were certified daily by gas chromatography/mass spectrometry (GC/MS) analysis for TO-14 target compounds. If the canister failed, all three canisters from that particular batch were evacuated and re-cleaned.

After air sampling at the site was completed, the canisters were either shipped or delivered in person to the Air Toxics LTD. laboratory for GC/MS analysis along with the necessary chain-of-custody documentation. Upon receiving the canisters, a pressurization process was performed by the laboratory.

The pressurization process involved attaching the canisters to a manifold system where each canister was opened and a final pressure reading was taken from a gauge. A purge valve was opened and high purity nitrogen gas entered each of the open canisters so that a pressure of 5 psi (equal to 6 liters in volume) was achieved. The purge gas and the canister were then closed and a brass plug was placed on the canister. The final pressure was recorded and the volume of dilution gas that was added was accounted for in the final calculations.

Field Sampling

A number of procedures were followed routinely to ensure that proper field sampling techniques were performed. Upon receipt of the canisters from the laboratory at the site, the canisters were

checked for damage during shipping and tested for leaks. A number of different quality control samples were taken and the frequency and sample types are described in the following sections.

Field-Split Duplicate Samples

Duplicate samples were collected to show precision and reproducibility. A duplicate sample was collected by sampling air from one port (the inlet) and splitting the air line to two different Summa® canisters. Results of the duplicate analyses for TO-14 and TO-11 are shown (Tables 1 and 2). For the duplicate TO-14 and TO-11 analyses, relative percent difference between the two duplicate samples was 10 % or less for each component of the matrix.

Table 1. Duplicate TO-14 sample analysis, relative percent difference, and trip blank TO-14 analysis (values for individual compounds in ppbv).

Sample #	**154**	**154-QAD**	**Relative % Difference**	**158-QAT**
Date	**12/23/97**	**12/23/97**	**12/23/97**	**12/23/97**
Freon 12	510	480	5.9	U
VC	900	840	6.7	U
1,1-DCE	7500	7200	4.0	U
Freon 113	360	350	2.8	U
DCM	1800	1700	5.6	U
1,1-DCA	2700	2500	7.4	U
cis-1,2-DCE	2800	2700	3.6	U
1,1,1-TCA	30000	29000	3.3	U
1,2-DCA	200	180	10.0	U
TCE	18000	18000	0.0	U
Toluene	7600	7400	2.6	U
1,1,2-TCA	160	160	0.0	U
PCE	20000	19000	5.0	U
Chlorobenzene	340	320	5.9	U
Ethylbenzene	530	500	5.7	U
m/p-xylene	2300	2200	4.3	U
o-xylene	1200	1100	8.3	U
1,3,5-TMB	1800	1700	5.6	U
1,2,4-TMB	6200	6000	3.2	U
1,3-DCB	1100	1000	9.1	U
1,4-DCB	2800	2600	7.1	U
1,2-DCB	14000	14000	0.0	U
Acetone	1000	930	7.0	U
4-Ethyltol.	2600	2500	3.8	U

Table 2. Duplicate TO-11 sample analysis, relative percent difference, and trip blank TO-11 analysis (values for individual compounds in ppmv referenced to heptane).

Sample #	N154	N155QAD	Relative % Difference	N158QAT
Date	**12/23/97**	**12/23/97**	**12/23/97**	**12/23/97**
TMNOC	400	390	2.5	0.02

Trip Blanks

A trip blank sample involved taking a Summa® canister that was sent to the site from the laboratory and returning it to the laboratory for analysis. Trip blanks were sent to assess possible contamination at the site and during transit. The results for the trip blank analyses are shown in Tables 1 and 2. The TO-14 trip blank analysis demonstrated no contamination of the sample while the TO-11 sample contamination was minimal (near detection limits).

Analytical

Within the laboratory, a number of analytical quality control procedures and measurements were taken. These included the measurement of analytical blanks, surrogate recovery, and spike analyses.

Instrument Calibration

A five point internal standard calibration was performed using standards dynamically blended from commercial stock. In general, all calibration standards were National Institute of Standards and Technology (NIST) traceable. By varying flow rates through a calibration gas manifold using digital flow sensors (1% accuracy), the blending of balance gas and calibration gas was performed. The blends were stored in Summa® canisters and held for up to one month. The balance gas was ultrahigh purity nitrogen.

The calibration curves of the instrument were verified daily by running a mid-point standard. The response could not vary by more than ± 30 % difference for 90% of the TO-14 compound list or re-calibration was performed.

Analytical Blanks

At a minimum, analytical blanks were run daily using clean zero reagent blank air. Reagent blanks were run on the GC/MS prior to the analysis of samples and after the analysis of standards. At a minimum, all analytes must have been less than their specific detection limits. If contamination was detected, the instrument was cleaned and a second blank was analyzed. Samples were not analyzed until a clean blank was obtained. The results for the TO-14 and TO-11 analysis demonstrated that all analytical blanks showed no detection of any analytes (Table 3).

Table 3. Analytical blank TO-14 and TO-11 sample analyses for selected target compounds and TNMOC.

Date	12/3/97	12/23/97	1/13/98	1/30/98	2/27/98	4/3/98
VC	U	U	U	U	U	U
cis-1,2-DCE	U	U	U	U	U	U
1,1 - DCA	U	U	U	U	U	U
1,1,1 - TCA	U	U	U	U	U	U
TCE	U	U	U	U	U	U
Toluene	U	U	U	U	U	U
PCE	U	U	U	U	U	U
TNMOC	U	U	U	U	U	NM

Surrogate Recovery

Internal surrogates were passed through the GC/MS instrument along with the sample. The surrogate percent recovery is a measure of analytical accuracy and has method limits of 70-130 % recovery (Table 4). For the TO-14 analyses, surrogate recoveries for all samples analyzed were within ± 10 %.

Table 4. Surrogate recovery efficiencies for TO-14 samples. Method recovery limits are 70-130 %.

Sample #	N141	N142	N144	N154	N155QAD	N156	N157	N158QAT
Date	12/3/97	12/3/97	12/3/97	12/23/97	12/23/97	12/23/97	12/23/97	12/23/97
Octafluorotoluene	103	99	99	101	95	104	104	101
Toluene-d8	102	100	101	97	98	96	98	93
4-Bromofluorobenzene	113	111	110	110	110	111	108	99

Sample #	N213	N214	N215	N212	N295	N297	N296	
Date	1/13/98	1/13/98	1/13/98	1/13/98	1/30/98	1/30/98	1/30/98	
Octafluorotoluene	107	99	104	102	103	104	99	
Toluene-d8	98	99	98	99	93	95	98	
4-Bromofluorobenzene	110	111	109	103	108	108	104	

Sample #	N347	N348	N349	N455	N456	N457		
Date	2/27/98	2/27/98	2/27/98	4/3/98	4/3/98	4/3/98		
Octafluorotoluene	100	103	102	100	103	99		
Toluene-d8	93	100	101	102	102	102		
4-Bromofluorobenzene	108	104	105	101	102	102		

Spike Analysis

This analysis was performed for the TO-14 and TO-11 methods to determine the effect of the matrix on the compounds of interest. Spike recovery data was within ± 20 % for the seven target compounds (Table 5) for the TO-14 analysis. However, for the TO-11 analysis, the spike recovery was out of this range for three out of the four sampling events.

Table 5. Method spike recovery efficiencies for TO-14 and TO-11 analysis.

Date	12/3/97	12/23/97	1/13/98	1/30/98	2/27/98
VC	95	101	107	116	106
cis-1,2-DCE	96	99	105	117	101
1,1 - DCA	97	102	106	118	98
1,1,1 - TCA	92	99	105	114	101
TCE	96	101	102	112	102
Toluene	99	103	102	117	94
PCE	89	93	90	105	106
TNMOC	128	92	73	71	NM

On-Site Air Sampling

On-site air samples were obtained using Tedlar™ bag grab samples. These samples were taken simultaneously from individual ports along the system and the gas samples were injected into an on-site GC for analysis. Additionally, samples of air were drawn into a photoionization detector (PID) for rapid assessment of contaminant concentrations. Numerous types of quality control samples were taken and the frequency and sample types are described in the following sections.

Analytical

Analytical quality control samples included calibration check standards, blank samples, and sampling duplicates.

Instrument Calibration

An initial calibration curve based on a known standard of a gas mixture was created. Using a 10-ml gas syringe, a known volume of a known concentration of gas was injected into the GC. An area count was measured and recorded. The known concentration of gas was then diluted by 25% and injected into the GC to establish the second point of the curve. This process was repeated until a five-point calibration curve was established.

For the PID, a zero gas and a gas of known concentration were injected into the instrument to establish a two-point calibration curve.

Calibration Check Standards

Weekly calibration checks (using a mid-point calibration standard) were performed to assess the accuracy of the calibration curve and to assess any drift with the instrument (Table 6). If more than ± 20 % drift was seen for any component of the mixture, a second calibration check standard was measured. If the range was exceeded for any of the components for this second check standard, a new calibration curve was created. On 1/27/98, a new curve was needed as the calibration standard was out of range. All other calibration checks were satisfactory.

Table 6. Calibration check standards for on-site GC analysis. Percent recoveries must be within ± 20 % otherwise a new calibration curve is developed.

	11/26/97	**12/3/97**	**12/23/97**	**1/12/98**	**1/15/98**
Sample #	**100CAL-V-N111**	**100CAL-V-N119**	**100CAL-V-N150**	**100CAL-V-N185**	**100CAL-V-N207**
1,2-DCE	91.00	99.60	97.64	95.58	87.20
1,1 - DCA	90.00	99.10	99.02	76.90	87.20
1,1,1 - TCA	89.00	99.10	95.08	99.80	86.50
TCE	84.00	98.20	90.46	83.11	85.20
Toluene	50.00	97.30	82.32	89.17	84.60
PCE	82.00	99.90	76.50	97.78	80.50

	1/23/98	**1/27/98**	**2/2/98**	**2/27/98**	**3/6/98**
Sample #	**100CAL-V-N233**	**100CAL-V-N262**	**100CAL-V-N289**	**100CAL-V-N318**	**100CAL-V-N348**
1,2-DCE	93.20	84.40	93.30	98.90	91.70
1,1 - DCA	84.50	**68.00**	84.70	96.40	98.00
1,1,1 - TCA	97.10	85.40	94.60	97.40	90.60
TCE	91.10	**51.00**	82.00	91.90	99.20
Toluene	97.30	**32.90**	80.30	94.20	98.20
PCE	93.10	**18.30**	80.00	88.00	95.20

	3/20/98	**4/3/98**	**4/6/98**	**4/10/98**	
Sample #	**100CAL-V-N407**	**100CAL-V-N423**	**100CAL-V-N459**	**100CAL-V-N467**	
1,2-DCE	97.60	97.20	95.40	96.40	
1,1 - DCA	86.50	85.40	84.10	99.30	
1,1,1 - TCA	99.00	98.70	96.90	96.60	
TCE	80.00	85.30	79.00	95.80	
Toluene	77.00	85.10	76.80	96.00	
PCE	78.50	77.20	79.00	94.60	

In order to establish the accuracy of the calibration gas itself, a sample of the gas was sent to an off-site sub-contracted laboratory (Air Toxics LTD.) for TO-14 analysis. In addition, a sample of the gas was sent to the supplier of the gas cylinder (Praxair) for analysis. The findings from Air Toxics LTD. demonstrated agreement with the listed concentrations within 20 % while Praxair's findings were within 8 % (Table 7). From these results, a new calibration curve was established with the basis of the data reflecting the findings established by Air Toxics LTD. Air Toxics LTD. results were used because all outside air analysis was being conducted by their organization and the quality of the on-site air results were to be eventually compared with those findings from Air Toxics LTD.

Table 7. Calibration gas quantification study.

	Original Reported	**Air Toxics Reported**		**Praxair Reported**	
Compound	**Concentration**	**Concentration**	**Percentage**	**Concentration**	**Percentage**
	(PPM)	**(PPM)**	**(%)**	**(PPM)**	**(%)**
Vinyl Chloride	-	-	-	-	-
1,2-Dichloroethylene	10.1	8.3	82.2	9.7	96.0
1,1-Dichloroethane	10.1	8.8	87.1	9.9	98.0
1,1,1-Trichloroethane	39.7	34.0	85.6	37.8	95.2
Trichloroethylene	39.9	32.0	80.2	36.7	92.0
Toluene	14.9	13.0	87.2	13.9	93.3
Perchloroethylene	40.1	34.0	84.8	38.7	96.5
1,2-Dichlorobenzene	19.8	18.0	90.9	19.5	98.5

For the PID instrument, calibration check standards were performed prior to sampling of field samples. Results of the calibration check demonstrated an agreement with the check standard within ± 10 % (Table 8). If the calibration were out of this range, the instrument would be recalibrated.

Table 8. Calibration check standard results and blank air readings for the PID instrument.

Date	Time	Ambient (ppmv)	% Calibration (100% @60)	Date	Time	Ambient (ppmv)	% Calibration (100% @60)
11/7/97	10:00	0.70	90.00	1/23/98	10:00	1.50	95.00
11/12/97	8:20	1.60	90.00	1/26/98	10:00	1.00	98.00
11/12/97	9:00	1.20	90.00	1/27/98	10:15	1.30	98.00
11/12/97	11:20	5.20	90.00	1/28/98	10:30	1.20	97.00
11/12/97	12:00	8.40	90.00	1/29/98	10:30	1.40	97.00
11/12/97	15:20	6.50	90.00	1/30/98	10:15	1.70	93.00
11/12/97	16:20	5.80	90.00	2/24/98	10:00	0.80	100.00
11/13/97	9:40	3.40	90.00	2/25/98	10:30	0.90	100.00
11/14/97	9:30	4.80	90.00	2/26/98	13:30	1.80	100.00
11/18/97	9:25	5.60	95.00	2/27/98	11:00	1.10	100.00
11/19/97	14:50	6.50	95.00	3/2/98	11:00	1.20	100.00
11/21/97	15:00	4.30	92.00	3/3/98	11:00	1.50	100.00
11/21/97	15:40	8.80	92.00	3/4/98	11:00	1.70	100.00
12/18/97	10:40	0.60	100.00	3/5/98	14:00	1.70	100.00
12/18/97	17:00	1.30	100.00	3/6/98	11:00	2.00	91.00
12/22/97	10:10	0.90	100.00	3/9/98	11:00	1.60	95.00
12/23/97	16:40	0.90	93.40	3/10/98	10:30	1.10	98.00
12/24/97	10:00	1.10	93.40	3/19/98	13:30	1.10	92.00
12/24/97	14:45	1.40	93.40	3/20/98	10:30	1.30	94.00
12/29/97	10:30	1.90	100.00	3/30/98	10:30	0.70	96.00
12/30/97	12:00	1.00	100.00	3/31/98	10:30	0.90	96.00
1/9/98	9:30	0.70	98.30	4/1/98	10:30	1.10	97.00
1/12/98	15:00	0.70	93.30	4/2/98	10:30	1.10	96.00
1/13/98	10:30	1.20	94.10	4/3/98	14:30	1.30	95.00
1/14/98	10:30	0.80	99.20	4/6/98	10:30	1.10	96.00
1/15/98	10:30	1.10	94.50	4/7/98	10:30	1.60	94.00
1/16/98	10:30	1.20	90.00	4/8/98	10:30	1.50	98.00
1/19/98	10:00	1.10	98.00	4/9/98	10:30	1.60	97.00
1/20/98	11:30	1.30	97.00	4/10/98	10:30	1.50	96.00
1/21/98	11:15	1.10	95.00	4/13/98	12:30	1.30	97.00
1/22/98	10:30	1.50	100.00	4/14/98	10:30	1.40	95.00

Blank Samples

The Tedlar™ bags used for gas collection were cleaned after every use by purging with nitrogen gas to ensure contamination was kept to a minimum. After purging, one bag was filled with ambient air and injected into the GC to quantify any contamination (Table 9). Only one sample, on 2/24/98, demonstrated some contamination.

Table 9. Sample blank analysis for on-site GC measurements for target compounds.

	11/14/97	11/20/97	11/25/97	11/26/97	12/1/97	12/22/97	1/9/98	1/12/98
Sample #	**Air-V-N54**	**Air-V-N71**	**Air-V-N97**	**Air-V-N106**	**Air-V-N124**	**Air-V-N152**	**Air-V-N190**	**Air-V-N203**
VC	U	U	U	U	U	U	U	U
1,2-DCE	U	U	U	U	U	U	U	U
1,1 - DCA	U	U	U	U	U	U	U	U
1,1,1 - TCA	U	U	U	U	U	U	U	U
TCE	U	U	U	U	U	U	U	U
Toluene	U	U	U	U	U	U	U	U
PCE	U	U	U	U	U	U	U	U

	1/13/98	1/14/98	1/15/98	1/20/98	1/21/98	1/22/98	1/23/98	1/26/98
Sample #	**Air-V-N208**	**Air-V-N218**	**Air-V-N228**	**Air-V-N243**	**Air-V-N247**	**Air-V-N253**	**Air-V-N258**	**Air-V-N265**
VC	U	U	U	U	U	U	U	U
1,2-DCE	U	U	U	U	U	U	U	U
1,1 - DCA	U	U	U	U	U	U	U	U
1,1,1 - TCA	U	U	U	U	U	U	U	U
TCE	U	U	U	U	U	U	U	U
Toluene	U	U	U	U	U	U	U	U
PCE	U	U	U	U	U	U	U	U

	1/30/98	2/2/98	2/24/98	2/25/98	2/27/98	3/3/98	3/6/98	3/19/98
Sample #	**Air-V-N290**	**Air-V-N298**	**Air-V-N330**	**Air-V-N335**	**Air-V-N343**	**Air-V-N363**	**Air-V-N374**	**Air-V-N408**
VC	U	U	U	U	U	U	U	U
1,2-DCE	U	U	U	U	U	U	U	U
1,1 - DCA	U	U	U	U	U	U	U	U
1,1,1 - TCA	U	U	**0.16**	U	U	U	U	U
TCE	U	U	**0.37**	U	U	U	U	U
Toluene	**0.06**	U	**0.03**	U	U	U	U	U
PCE	U	U	**0.06**	U	U	U	U	U

	3/20/98	3/30/98	3/31/98	4/1/98	4/2/98	4/3/98	4/7/98	4/10/98
Sample #	**Air-V-N413**	**Air-V-N425**	**Air-V-N430**	**Air-V-N436**	**Air-V-N446**	**Air-V-N451**	**Air-V-N468**	**Air-V-N487**
VC	U	U	U	U	U	U	U	U
1,2-DCE	U	U	U	U	U	U	U	U
1,1 - DCA	U	U	U	U	U	U	U	U
1,1,1 - TCA	U	U	U	U	U	U	U	U
TCE	U	U	U	U	U	U	U	U
Toluene	U	U	U	U	U	U	U	**0.08**
PCE	U	**0.03**	U	U	U	U	U	**0.09**

A sample of ambient air was directly injected into the PID to quantify any background contamination at the site (Table 8). Results for the blank air show minimal contamination after 11/21/97. Prior to this date, problems with the lamp in the instrument and the introduction of water into the sampling system caused suspect readings. The lamp and the sampling system were cleaned and the instrument was operated without any further problems.

Sampling Duplicates

In order to ensure the ability of the instrumentation to provide verifiable and consistent measurements, duplicate samples were analyzed using two different samples per sampling bag (Table 10). The results show good repeatability of the data as reported by the on-site GC.

Table 10. Sampling duplicates taken for the target compounds for on-site GC analysis. Values in ppmv.

Sample #	BFI-V-N134	BFI-V-N135	Relative % Difference
VC	1.39	1.43	3.00
1,2-DCE	1.90	1.92	1.15
1,1 - DCA	4.50	4.50	0.00
1,1,1 - TCA	8.78	8.87	0.98
TCE	13.94	14.13	1.35
Toluene	10.63	10.48	1.41
PCE	13.94	13.80	1.02

Sample #	CSTRI-V-N132	CSTRI-V-N133	Relative % Difference
VC	1.37	1.35	1.90
1,2-DCE	1.88	1.83	2.77
1,1 - DCA	4.41	4.31	2.13
1,1,1 - TCA	8.76	8.54	2.55
TCE	13.75	13.39	2.58
Toluene	10.55	10.25	2.78
PCE	13.43	13.43	0.06

Sample #	CSTRE-V-N130	CSTRE-V-N131	Relative % Difference
VC	1.31	1.33	1.73
1,2-DCE	1.87	1.91	2.14
1,1 - DCA	4.28	4.30	0.51
1,1,1 - TCA	8.61	8.81	2.27
TCE	13.45	13.71	1.88
Toluene	10.13	10.32	1.77
PCE	13.19	13.55	2.66

Laboratory Preventive Maintenance

Preventive maintenance was performed according to the procedures set forth in the analytical equipment operating manuals. The carrier and calibration gas cylinders were checked weekly for leaks. Instruments were checked and repaired if the quality of the data began to degrade significantly. Significant degradation in data was interpreted to be any large shift in calibration curves, decrease in sensitivity, or degradation in peak resolution. All instrument problems and corrective actions taken were documented and recorded in a field logbook.

Off-Site Water Sampling and Analysis Protocol

Water analyses were performed by Sequoia Analytical (Sacramento, CA) using methods SW8260 (VOCs), EPA 405.1 (BOD_5), EPA 410.1 (COD), EPA 160.2 (TSS/VSS), EPA 160.1 (TDS), and EPA 300 (chloride). All containers for sampling were obtained from Sequoia Analytical prior to the sampling event. Sequoia Analytical purchased containers in large lots from various commercial sources that were equivalent, in terms of construction materials and cleaning protocols, to those listed in the Federal Register, October 26, 1984 and the most current version of SW-846. EPA SW-846 is the *Test Methods for Evaluating Solid Waste: Physical and Chemical Methods.*

Bottles for analyses were purchased by Sequoia Analytical from suppliers who, upon request, certified that the containers had been cleaned by protocols prescribed in the various EPA methods. Containers were prepared in a designated area according to posted Standard Operating Procedures. Next, containers were clearly marked to indicate the preservative added, given a sample description label, and then stored in an orderly fashion.

After collection of samples, the samples were placed in a cooler with blue ice and stored temporarily until returned to the laboratory. For those samples where it was appropriate, a trip blank was prepared before sampling that accompanied the bottles out to the sampling site, during the sampling process, and back to the laboratory in the same cooler.

All collected samples were delivered in person to the Sacramento office of Sequoia Analytical along with the necessary chain-of-custody documentation. Because CLP packages were required, all samples were shipped via courier from the Sacramento Laboratory to the Redwood City laboratory. When the samples were received at the Redwood City laboratory, the personnel in Sample Control checked to ensure that all samples listed on the *Chain of Custody* were, in fact, present and in satisfactory condition. The temperature of samples in the cooler were taken with a laser gun thermometer and documented on the *Chain of Custody*. They signed and dated the *Chain of Custody* form and supplied each sample container a unique sample number and stored them appropriately in cold storage or at room temperature. Each of these unique sample numbers were entered into a computer network along with the client's name, the tests requested, hold times, the turnaround status, condition of the sample, and any special comments.

Field Sampling

After pickup of the proper containers from Sequoia Analytical, sampling for various parameters commenced. In conjunction with the field samples, quality control samples were collected to ensure reproducibility and consistency of the data.

Collection Duplicates

Duplicate samples were analyzed to evaluate the precision of the analytical process. Duplicate water samples were collected by sampling two separate samples from one port in series. Results of the duplicate analyses for methods SW8260 (VOCs), EPA 405.1 (BOD_5), EPA 410.1 (COD), EPA 160.2 (TSS/VSS), EPA 160.1 (TDS) are shown (Tables 11 and 12). For the duplicate analysis for the SW8260 method, the relative percent difference between the two duplicate samples for each component of the matrix was less than 15 %. For the methods EPA 405.1 (BOD_5), EPA 410.1 (COD), the EPA 160.2 (TSS/VSS), the relative percent difference was less than 10 % for duplicate samples. However, for the method EPA 160.1 (TDS), the relative percent difference was greater than 50 %, making these results suspect.

Table 11. Collection duplicate and trip blank results for SW8260 analysis. Results are given in ppbv.

Sample #	**CSTR-N353**	**CSTR-N353-QAD**	**Relative % Difference**	**QAT**
Date	**3/2/98**	**3/2/98**	**3/2/98**	**3/9/98**
1,2-DCB	140	120	14.30	U
1,3-DCB	6.9	6.1	11.59	U
1,4-DCB	20	18	10.00	U
1,1DCA	9.1	9	1.10	U
1,2-DCA	2.1	2.1	0.00	U
1,1-DCE	2.7	3.1	12.90	U
cis-1,2-DCE	7.6	7.2	5.26	U
DCM	7.1	7.4	4.05	U
PCE	27	28	3.57	U
1,1,1-TCA	34	35	2.86	U
1,1,2-TCA	3.5	3.5	0.00	U
TCE	31	32	3.13	U
1,3,5-TMB	3	3.1	3.23	U

Table 12. Collection duplicate results for methods EPA 405.1 (BOD_5), EPA 410.1 (COD), EPA 160.2 (TSS/VSS), and EPA 160.1 (TDS). Results are given in ppm.

Sample #	N355	N355QAD	Relative % Difference	N356	N356QAD	Relative % Difference
Date	**3/2/98**	**3/2/98**	**3/2/98**	**3/2/98**	**3/2/98**	**3/2/98**
Chloride	NM	NM	NA	58	56	3.45
TDS	NM	NM	NA	4150	1839	55.69
TSS	NM	NM	NA	2210	2310	4.33
TVS	NM	NM	NA	NM	NM	NA
COD	NM	NM	NA	NM	NM	NA
BOD	120	110	8.33	NM	NM	NA

Trip Blanks

Trip blanks were prepared by the analytical laboratory with purified water and were sent along with the sample containers to indicate the presence of contamination from handling errors or cross contamination. Results for a trip blank sample for SW8260 analysis were all undetected for each component of the matrix (Table 11).

Field Blanks

Field blanks were collected to evaluate the field conditions that may contribute to sample contamination. These field blanks were equivalent to obtaining a background reading at the sampling site and were collected at a sample location at the time of field sampling. Field blanks were obtained for methods EPA 410.1 (COD), EPA 160.2 (TSS/VSS), and EPA 160.1 (TDS) (Table 13). From the results for all methods, minimal contamination occurred because of field conditions.

Table 13. Field blank results (QAF) for methods EPA 410.1 (COD), EPA 160.2 (TSS/VSS), and EPA 160.1 (TDS). Results are given in ppm.

Sample #	N478	N478QAF	N479	N479QAF	N480	N480QAF
Date	**4/8/98**	**4/8/98**	**4/8/98**	**4/8/98**	**4/8/98**	**4/8/98**
Chloride	NM	NM	NM	NM	NM	NM
TDS	NM	NM	5700	16	NM	NM
TSS	NM	NM	4600	U	NM	NM
TVS	NM	NM	NM	NM	6000	U
COD	820	37	NM	NM	NM	NM

Analytical

Instrument Calibration

Initially, each instrument was calibrated for the analytical method for which it was allocated. Once the operating parameters were established according to that method, the analyst prepared standard solutions containing all the analytes of interest, any internal standards, and any surrogate standards appropriate to the method. To establish the calibration curve for a particular analyte, these standard solutions were prepared at graduated dilutions. One of the concentrations was at or just above the reporting limit while the others defined the working range for the instrument.

Instrument calibrations were performed on an as needed basis in accordance with the specific method requirements for organics and inorganics instrumentation. Calibration curves were generated when fundamental changes to the instrumentation occurred or when results of QC Check Standards indicated the system was not operating within acceptance limits.

Calibration factors (CF) were calculated for those methods that used external standards while the response factors (RF) were calculated for those methods that used internal standards. The CF's or RF's were tabulated for each of the five concentrations for each of the analytes and surrogates. The five CF's or RF's for each analyte or surrogate had a Percent Relative Standard Deviation (%RSD) within acceptance limits in order for the laboratory to use the average RF or CF. Otherwise the actual curve was used. The validity of the calibration curve was checked daily for most instruments and more frequently for instruments with particularly sensitive detectors that tended to drift. The analyst prepared a daily calibration check standard solution in the same manner as for the initial calibration standard solutions. The daily calibration check standard solution CF or RF was required to be within the acceptance limits of the average CF or RF of the calibration curve.

Method Blanks

The method blank estimated the analytical response attributable to all factors other than the analyte in the sample. Method blanks were analyzed identically to samples and were prepared from laboratory matrices that did not contain analytes.

Any analytes found in a method blank should have been present at a level less than the 'Reporting Limit' for the analytes of interest. If not, the source of contamination was investigated and corrective action developed. Essentially, all liquid samples demonstrated no detection of analytes (Tables 14 and 15).

Table 14. Method blank results for SW8260 analysis.

Date	12/29/97	1/14/98	1/28/98	3/2/98	3/9/98	4/15/98
1,2-DCB	U	U	U	U	U	U
1,3-DCB	U	U	U	U	U	U
1,4-DCB	U	U	U	U	U	U
1,1DCA	U	U	U	U	U	U
1,2-DCA	U	U	U	U	U	U
1,1-DCE	U	U	U	U	U	U
cis-1,2-DCE	U	U	U	U	U	U
DCM	U	U	U	U	U	U
PCE	U	U	U	U	U	U
1,1,1-TCA	U	U	U	U	U	U
1,1,2-TCA	U	U	U	U	U	U
TCE	U	U	U	U	U	U
1,3,5-TMB	U	U	U	U	U	U

Table 15. Method blank results for EPA 405.1 (BOD_5), EPA 410.1 (COD), EPA 160.2 (TSS/VSS), and EPA 160.1 (TDS). Results are given in ppm.

Date	12/29/97	1/6/98	1/14/98	1/19/98	1/28/98	3/2/98	3/9/98	4/1/98	4/8/98
Chloride	U	0.67	U	U	U	U	NM	U	NM
TDS	U	U	U	U	U	U	U	U	U
TSS	U	U	U	U	U	U	U	U	U
TVS	U	U	U	U	U	U	U	U	U
COD	U	U	U	U	U	U	U	U	U
BOD	NM	NM	NM	NM	NM	U	NM	U	NM

Surrogate Recovery

Surrogate compounds were spiked into all environmental samples being analyzed for SW8260. Because surrogates were spiked prior to processing, they served as a check on the efficiency of extraction. The percent recovery of surrogates was documented and compared to established control limits. The use of surrogates ensured that all environmental samples went through the analytical process with acceptable recovery. Should a sample have failed the established control limits, it was re-extracted and re-analyzed. If the recovery failed the established control limits a second time, the data was flagged and reported as an estimated or minimum value. Surrogate recovery efficiencies for SW8260 analyses were within the control limits that ranged from 65 to 120 % (Table 16).

Table 16. Surrogate recovery efficiencies for SW8260 analyses.

Sample #	CSTR-N143	CSTR-N171	BF-N172	CSTR-N222	CSTR-N283
Date	**12/3/97**	**12/29/97**	**12/29/97**	**1/14/98**	**1/28/98**
1,2-Dichloroethane-d4	94	108	111	102	100
Toluene-d8	103	98	97	102	108
4-Bromofluorobenzene	97	100	102	100	103

Sample #	CSTR-N353	CSTR-N353-QAD	QAT	CSTR-N502
Date	**3/2/98**	**3/2/98**	**3/9/98**	**4/15/98**
1,2-Dichloroethane-d4	98	95	94	90
Toluene-d8	104	100	101	107
4-Bromofluorobenzene	100	96	95	101

Matrix Spike (MS)

The samples were spiked with known quantities of stable constituents that were representative of the target analytes of interest. The matrix spike was used to document the bias of a method in a given sample matrix. Matrix spike analyses (and matrix spike duplicate) were performed for methods SW8260, EPA 410.1 (COD), EPA 160.1 (TDS), and EPA 300 (chloride) (Tables 17 and 18). Results for SW8260 were within control limits. Results for EPA 160.1 (TDS) were generally within the control limits with the exception of samples taken 1/6/98 and 1/28/98. Results for methods EPA 410.1 and EPA 300 were generally out of the control limits. Hence, these results are flagged.

Matrix Spike Duplicate (MSD)

Split samples were spiked by the laboratory with identical concentration of target analyte(s) as the matrix spike. The matrix spike duplicates were used to document the precision and bias of the method in a given sample matrix (Tables 17 and 18). In addition to providing information concerning the effect of sample matrix, the duplicate spiked data can also provide an estimate of analytical precision for each spiked analyte. The component of variability represents the variability that occurred during chemical analysis of the samples. Results for SW8260 were within control limits. Results for EPA 160.1 (TDS) were generally within the control limits with the exception of samples taken 1/6/98 and compared well with the matrix spike results. Results for methods EPA 410.1 and EPA 300 were generally out of the control limits but did compare well with the matrix spike results. Hence, these results demonstrate that precision was obtainable using these methods.

Laboratory Control Sample

A volume of reagent water was spiked with known quantities of target analytes and required surrogates (for organics methods). Aqueous control samples were analyzed using the same sample preparation, reagents, and analytical methods employed for the environmental samples received. Laboratory control samples were performed for methods SW8260, EPA EPA 410.1 (COD), EPA 160.1 (TDS), and EPA 300 (chloride) (Tables 17 and 18). Results were within control limits for all analyses.

Table 17. Matrix spike (MS), matrix spike duplicate (MSD), and laboratory control sample (LCS) recoveries for SW8260 analyses of selected target compounds.

12/29/97	MS %	MSD %	LCS %	MS Limits	LCS Limits
1,1 DCE	92	88	88	60-140	65-135
TCE	98	100	94	60-140	70-130
Benzene	99	99	95	60-140	70-130
Toluene	97	98	95	60-140	70-130
Chlorobenzene	96	96	91	60-140	70-130

1/14/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
1,1 DCE	98	98	94	60-140	65-135
TCE	96	94	90	60-140	70-130
Benzene	94	94	90	60-140	70-130
Toluene	94	92	88	60-140	70-130
Chlorobenzene	92	90	88	60-140	70-130

1/28/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
1,1 DCE	80	86	90	60-140	65-135
TCE	88	96	100	60-140	70-130
Benzene	98	104	110	60-140	70-130
Toluene	94	100	104	60-140	70-130
Chlorobenzene	94	98	100	60-140	70-130

3/2/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
1,1 DCE	80	82	82	60-140	65-135
TCE	92	94	94	60-140	70-130
Benzene	100	98	98	60-140	70-130
Toluene	88	92	94	60-140	70-130
Chlorobenzene	92	94	94	60-140	70-130

4/1/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
1,1 DCE	84	80	76	60-140	65-135
TCE	98	94	98	60-140	70-130
Benzene	98	96	92	60-140	70-130
Toluene	94	92	90	60-140	70-130
Chlorobenzene	98	100	100	60-140	70-130

4/15/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
1,1 DCE	84	84	82	60-140	65-135
TCE	92	92	96	60-140	70-130
Benzene	104	104	106	60-140	70-130
Toluene	88	90	96	60-140	70-130
Chlorobenzene	88	90	94	60-140	70-130

Table 18. Matrix spike (MS), matrix spike duplicate (MSD), and laboratory control sample (LCS) recoveries for EPA EPA 410.1 (COD), EPA 160.1 (TDS), and EPA 300 (chloride).

12/29/97	MS %	MSD %	LCS %	MS Limits	LCS Limits
Chloride	0	400	100	75-125	80-120
TDS	86	86	98	75-125	80-120
COD	137	127	93	75-125	80-120

1/6/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
Chloride	90	100	100	75-125	80-120
TDS	140	140	98	75-125	80-120
COD	70	62	120	75-125	80-120

1/14/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
Chloride	130	130	99	75-125	80-120
TDS	108	108	110	75-125	80-120
COD	137	127	93	75-125	80-120

1/19/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
Chloride	60	60	100	75-125	80-120
TDS	92	92	92	75-125	80-120
COD	40	80	96	75-125	80-120

1/28/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
Chloride	68	68	100	75-125	80-120
TDS	72	82	90	75-125	80-120
COD	163	53	93	75-125	80-120

3/2/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
Chloride	50	50	97	75-125	80-120
TDS	95	78	90	75-125	80-120
COD	120	110	100	75-125	80-120

4/1/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
Chloride	110	110	97	75-125	80-120
TDS	90	86	98	75-125	80-120
COD	70	70	110	75-125	80-120

4/8/98	MS %	MSD %	LCS %	MS Limits	LCS Limits
TDS	101	105	100	75-125	80-120
COD	61	71	82	75-125	80-120

Matrix Duplicate

A split sample was prepared in the laboratory which was used to document the precision of a method in a given sample matrix. The matrix duplicate indicated within-run precision between the original sample and duplicate sample values in terms of relative percent difference (RPD). Matrix duplicates were performed for methods EPA 160.2 (TSS/VSS) and EPA 405.1 (BOD_5) (Table 19). All relative percent difference values were within 20 % with one exception on 4/8/98 for TSS.

Table 19. Matrix duplicate samples for EPA 160.2 (TSS/VSS) and EPA 405.1 (BOD_5). TSS = total suspended solids, VSS = volatile suspended solids, BOD = biochemical oxygen demand.

Date	TSS 1	TSS 2	Relative % Difference	VSS 1	VSS 2	Relative % Difference	BOD 1	BOD 2	Relative % Difference
12/29/97	990	830	16.16	810	910	10.99	NM	NM	NA
1/6/98	42	38	9.52	290	350	17.14	NM	NM	NA
1/14/98	95	100	5.00	370	420	11.90	NM	NM	NA
1/19/98	128	114	10.94	370	420	11.90	NM	NM	NA
1/28/98	30	29	3.33	3300	3000	9.09	U	U	NA
3/2/98	2310	2260	2.16	3900	3900	0.00	14	13	7.14
4/1/98	300	310	3.23	6480	6480	0.00	NM	NM	NA
4/8/98	150	200	25.00	6030	6200	2.74	NM	NM	NA

On-Site Water Sampling

On-site water samples were collected into glass beakers and immediately analyzed per manufacturer's instructions in the on-site laboratory. Analytical procedures involved properly calibrating the instruments and analyzing blank samples.

Analytical

Instrument Calibration

Instruments were checked regularly with known standards to assess the drift of the instrument. All instruments were calibrated according to manufacturer's instructions and check standards were run before each sample to ensure the calibration was correct.

Blank Samples

Before analyzing a water sample, blank samples consisting of distilled water were used to zero out all instruments and to establish a baseline value for each analytical method. If a blank sample demonstrated contamination, the sample container was cleaned and a new blank sample was analyzed.

Laboratory Preventive Maintenance

Preventive maintenance was performed according to the procedures set forth in the analytical equipment operating manuals. All equipment was monitored for unusual or anomalous readings. If shifts from the check standards occurred, new calibrations were initiated using solutions of known composition and concentration. All instrument problems and corrective actions taken were documented and recorded in a field logbook.